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**Editors:** B. Michel, T. Winkler,  
M. Werner, H. Fecht



# **ABSTRACTS**

**3rd**

**International Conference and Poster Exhibition**

# **Micro Materials**

# **MICROMAT 2000**

**April 17-19, 2000**

**Berlin**

**Germany**

**Sponsoring Societies**

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• IMAPS • FhG • DFG • AFM • DGzFP • AMA  
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**in cooperation**

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## Preface

The Micro Materials 2000 is the third congress organized by the Fraunhofer Institute IZM together with the German Association for Materials Research and Testing (DVM) in the field of materials research and materials applications to microsystem technology and microelectronics. After the very successful conferences in 1995 and 1997 this conference will be held again on an international level. In response to the call for papers the International Program Committee received more than 350 papers submitted from 45 countries. The understanding of materials problems in microelectronics and microsystem technologies applications has advanced considerably in recent years. But there are many questions and new trends which need to be dealt with.

The aim of this third congress is to identify and highlight the progress achieved since the MicroMat '97 concerning mainly the different fields of micro materials applications and bring together specialists from all over the world to discuss the recent trends. Because there has been observed a considerable interest in micromaterials characterization the MicroMat organizers have invited outstanding scientists and engineers to deliver plenary and key-note lectures in different fields of materials testing and simulation.

In addition to the plenary lectures, invited session and key-note session lectures, the program covers various topics of the conference with 260 oral and about 70 poster presentations.

This congress will be of interest to researchers and engineers from companies, from research institutes and universities who are working on various problems of materials applications in microtechnologies and microelectronics. The conference topics cover the fields of materials applications in automotive electronics, micromechatronics, high temperature electronics, semiconductor and telecommunication industries and other branches applying advanced microtechnologies.

On behalf of the Program Committee we would like to wish you a successful conference - and enjoy Berlin!

H. Reichl  
Head of the Fraunhofer  
Institute IZM Berlin

B. Michel H.-J. Fecht M. Werner  
Conference Chairmen

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## Sponsoring Societies of MicroMat 2000

- DVM** German Association for Materials Research and Testing
- Working Group Microsystem Technology
  - Working Group Micro- and NanoProbe Techniques
- IEEE/CPMT** Components, Packaging and Manufacturing Society
- ASME** The American Society of Mechanical Engineers
- RSJ** Robotics Society of Japan
- EU** European Commission, Information Society
- HITEN** European Network of High Temperature Electronics
- IMAPS** International Microelectronics and Packaging Society
- FhG** Fraunhofer-Gesellschaft e.V.
- DFG** Deutsche Forschungsgemeinschaft
- AFM** Association Francaise Mécanique
- DGzFP** German Society for Nondestructive Testing
- AMA** Fachverband für Sensorik e.V.
- ZVEI** Zentralverband Elektrotechnik und Elektronik Industrie  
Division for Electronic Components
- IRC** Innovation Relay Centre North Germany
- ISTLI** International Society for Technology, Law and Insurance
- ESIS** European Society for Structural Integrity
- SF2M** Société Française de Métallurgie et de Matériaux
- SEM** Society for Experimental Mechanics
- SVM** Saxon Society for Mechanics



**DVM**



DEUTSCHER VERBAND FÜR MATERIALFORSCHUNG UND -PRÜFUNG E.V.



EUROPEAN COMMISSION  
Industrie  
RTD: Information Technologies

## Scope & Sponsors

### Scope of Micro Materials 2000

Main fields of interest

- ☐ micro materials research
- ☐ micro materials testing
- ☐ micro materials simulation
- ☐ micro materials application

in microsystem technology and related fields of microcomponents and microsystems materials applications, micro mechatronics and MEMS.

### Sponsoring Companies and Institutions

The organizers also express their thanks to the following companies and institutions for their financial sponsoring and personal assistance:

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MicroMat at a Glance

Monday, April 17th

10.00 - 11.15	Opening Plenary Session I
11.30 - 12.45	Plenary Session II
14.00 - 14.50	Plenary Session III
15.00 - 15.50	Plenary Session IV
16.30 - 17.45	Semiplenary Sessions V
17.50 - 18.40	Semiplenary Sessions VI
	Semiplenary Sessions VII
19.30	Welcome Reception by the Governing Mayor of Berlin, E. Diepgen, in the Berliner Rathaus (Red Town Hall)

Tuesday, April 18th

	1	2	3	4	5	6	7	
08.30 - 10.40	Polymeric Materials	Damage and Failure Mechanisms	Micro- and NanoProbe Techniques	Nanomaterials	Packaging Materials	Special Applications	Electrical and Electronic Properties	
	8	9	10	11	12	13	14	
11.00 - 12.50	German Research Programs	Mechanical Properties I	Special Applications II	Ceramics and Glass	Packaging Materials II	Optical Methods I	Testing and Characterization I	

## Tuesday, April 18th

	15	16	17	18	19	20	21	
14.30 - 16.20	6M TV Tower Workshop	Microstructure and Material Properties	Mechanical Properties, Reliability	Thin Films and Layers	Physical Mechanisms and Methods I	Materials Characterization and Micromanufacturing	Sensor- and Actuator Materials	
	22	23	24	25	26	27	28	
17.00 - 18.50	Mechatronics, Mechatronics, MEMS Adaptions, MEMS	Advanced Methods and Applications I	Mechanical Properties II	Microlabration Micromachining Micromanufacturing	Testing and Characterization II	Optical Methods II	Special Applications III	
20.00 - 23.00	Cruise with Conference Dinner							

## Wednesday, April 19th

	29	30	31	32	33	34	35	36
08.30 - 10.40	Physical Mechanisms and Methods II	Diamond and Wide Bandgap Materials	Crack, Fracture, Fatigue	Simulation and Testing	High Temperat. Electronics and Thermal Problems	Packaging Materials and Encapsulation	Indentation Methods, Layers, Interfaces	Non-destructive Testing
	37	38	39	40	41	42	43	44
11.00 - 12.50	Special Applications IV	Diamond and Hard Coatings	Advanced Methods and Applications II	Stress, Strain, Reliability	Micromaterials and Environment	Packaging Materials - Modeling and Reliability	Bonding, Joining	Solder Materials
13.45 - 15.00	Plenary Session IX							
15.00 - 16.40	Plenary Session X							



**The abstracts are in alphabetic order  
of the first authors' names**

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## Effects of work-hardening characteristics on evaluating flow properties from indentation load-depth curve

Ahn, J.-H.; Jeon, E.-c.; Choi, Y.; Lee, Y.-H.; Kwon, D.

*School of Materials Science and Engineering, Seoul National University, Seoul, Korea*

Continuous indentation test is one of the most powerful techniques in evaluating the mechanical properties of micro-materials. In this study, the flow properties of materials were estimated by analyzing the indentation load-depth curve measured by continuous ball indentation test. In analyzing the indentation load-depth curve, both the elastic deflection and pile-up/sink-in behavior were taken into consideration. The amount of pile-up/sink-in was determined by the work-hardening exponent. Using the calculated contact radius between the indenter and the specimen at maximum load, the representative strain  $\epsilon_r$  beneath the indenter was obtained by analyzing the deformation behavior, which leads to the relation  $\epsilon_r = a \tan \gamma$ . Here,  $\gamma$  is the contact angle between the indenter and the specimen. And, the representative stress  $\sigma_r$  was obtained by using the relation  $P_m / \sigma_r = \Psi$  for fully plastic stage. Here,  $P_m$  is the mean contact pressure, and the constraint factor  $Y$  could be related to the work-hardening characteristics of material in this study. To obtain the work-hardening exponent, we performed the multiple indentation up to  $h/R=0.3$ , where  $h$  and  $R$  are indentation depth and indenter radius, respectively. Then, we could calculate the work-hardening exponent by using the Hollomon type flow equation. By iterative calculation, we optimized the value of work-hardening exponent. Various steels such as TMCP, SM50, SA508, SA213, SA106, and AISI1025, were tested. And, the flow properties of these materials derived from indentation test were compared to those from tensile test. The results showed that the present analysis could be successfully applied to predict the flow properties of structural steels.

## Flex, rigid, and ceramic CSP - lifetime behavior

*Albrecht, H.-J.\*; Jendry, J.\*\*; Müller, W.H.\*\*\*; Schwarz, B.\*; Teichmann, H.\*; Tilgner, R.\*\*\*\**

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This paper presents a detailed numerical, non-linear Finite Element (FE) analysis of three different CSPs which use PI flex, an Alumina ceramic, or a BT resin as interposer material. Lifetime predictions (number of cycles to failure) are carried out on the basis of equivalent creep strains accumulated during Thermal Cycle Tests (TCTs). Moreover, the development of temperature profiles under Power Cycle Tests (PCTs) is simulated and used to predict the resulting irreversible strains within the solder bumps. The predictions from FE simulations are compared to reliability experiments (TherMoire<sup>®</sup> displacement measurements, Weibull plots of board level reliability, and distributions of crack lengths). Finally, special consideration is given to an FE-study of the influence of the choice of the High-Density-Substrate (HDS) board and of its inner structure on the lifetime of the microelectronic component as a whole.



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## Using of charge density to calculate bulk modulus in semiconductors

*Al-Douri, Y.; Abid, H.; Aourag, H.*

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The cationic part of the electronic charge densities are obtained by using the empirical pseudopotential method (EPM) for tetrahedrally bonded semiconductors to calculate the bulk modulus. We have used EPM for determining suitable charge densities. In our pseudopotential approach we have adjusted the pseudopotential form factors by a nonlinear least-square method [1-3], in which all the parameters are simultaneously optimized under a definite criterion of minimizing the root-mean-square deviation. The empirical methods offer the advantage of applicability to a broad class of materials and illustrate trends. The calculated bulk modulus scale is in reasonable agreement with that of experimental and Cohen [4] results.

### References

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- [4] M. L. Cohen, Phys. Rev. B32, 7988, 1985.

## Quantitative contact spectroscopy by atomic-force acoustic microscopy

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*Fraunhofer-Institute for Nondestructive Testing, Saarbrücken, Germany*

In Atomic Force Microscopy (AFM) deflection of a microfabricated elastic beam with a sensor tip at its end is used to generate high-resolution images of surfaces. Dynamic modes, where the cantilever is vibrated while the sample surface is scanned, belong to the standard equipment of most commercial instruments. With a variety of these techniques, such as Force Modulation Microscopy, Ultrasonic Force Microscopy, Scanning Local Acceleration Microscopy, or Pulsed Force Microscopy, images can be obtained which depend on the elasticity of the sample surface. However, quantitative determination of Young's modulus of a sample surface with AFM is still a challenge especially when stiff materials such as hard metals or ceramics are encountered. In this contribution the evaluation of the cantilever vibration spectra at ultrasonic frequencies is presented in order to discern local elastic data quantitatively. With such an Atomic Force Acoustic Microscope (AFAM), the local contact stiffness  $k$  can be measured. When the contact area of the tip is known,  $k$  can be inverted to determine the local surface elasticity with a spatial resolution of approx. 20 nm. Experimental results on different piezoelectric ceramics are presented. In imaging, our technique allows to make the domains visible due to their elastic contrast and anisotropy.

Thin films of nanocrystalline ferrites with spinel structure can be used in different recording systems. An important mechanical parameter of such films are their elastic properties. The measurement of the Young's modulus of nanocrystalline film as a function of the oxidation temperature of 200 nm thick films were undertaken with AFAM and will be presented. It turns out that the coercivity is correlated with the chemical gradients induced by the thermal oxidation causing tensile stresses and compressive stresses in the film.

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## Influence of microstructure of electroless deposited Ni used as under bump metallization for high temperature solders

Anhöck, S.\*; Ostmann, A.\*; Nieland, C.\*\*; Auerswald, E.\*\*; Reichl, H.\*\*

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Electroless nickel deposits are widely used in microelectronics. Most suitable is the chemical nickel deposition which is extensively applied in printed circuit boards as a diffusion barrier on copper leads. On wafer level the electroless Ni plating has also gained a high interest especially for the flip chip technology where the Ni is used as under bump metallization (UBM). The Ni bumping process provides a selective autocatalytic metal deposition directly on the aluminum pads of wafers, no costly equipment for sputtering, photo lithography and electroplating is required.

Due to the nature of the chemical deposition process the plated layers consist of nickel and phosphorous using sodium hypophosphite as reducing agent. For the reduction of Ni ions to Ni atoms the hypophosphite delivers the electrons and decomposed into  $H_2$ ,  $H_2O$  and P, while deposition. The phosphorous is embedded in layers. Using the electroless nickel bumping process developed at the Fraunhofer IZM/TU Berlin a phosphorous content of about 9 wt.-% with a small variation of about 1 wt.-% can be measured. A high phosphorous content leads to an amorphous microstructure of the Ni bump. During a heat treatment up to 350 °C a crystallization into Ni and  $Ni_3P$  takes place. A change of physical, chemical and electrical properties results from this crystallization.

Besides the layers structure of the Ni/P bumps a vertical growth structure can be observed. Before electroless Ni deposition the Al pad an activation of the Al is essential. After removing Al oxide and organic residues a thin zinc seeding layer is coated on the Al pads. The zinc will be replaced by Ni while Ni deposition. Vertical lines separate different growth columns of Ni/P starting from the zinc grains. Even after 4  $\mu m$  Ni bump height the Ni/P layer is a compact layer because the columns are grown together. A mushroom shaped form of bump is achieved.

The microstructure resulting from the phosphorous content and the vertical growth structure influences the reaction with the solder and the reliability of the metallurgical system UBM/solder. Especially for high temperature solders such as PbSn5 the thermal activation during the reflow and the bonding process can change the microstructure of the bump.

The focus of the poster is to introduce the results from the investigations of microstructure of electroless Ni bumps. Different x-ray diffraction measurements and thermal analysis were carried out to characterize the changes in microstructures during and

after bonding using high temperature solders. In order to observe the phosphorous diffusion the microstructure was appeared by etching the polished surface at different temperatures and duration times. The surface and the column structure was also observed with atomic force microscopy. The influences of the microstructure will be described. The impact on the reliability of the metallurgical system Ni-UBM/solder will be discussed for high temperature applications.

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## Reliability of soldered Nickel bumps for high temperature application

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Electronic components are applied more and more at higher operation temperatures. For the reliability the most difficult problem is to find a suitable metallurgical systems, especially for temperatures above 200 °C. Typical difficulties which limit the life time of electronic components include the altered electric behavior due to interdiffusion of solder and metallization materials or the brittleness caused by intermetallic phase formation. So, the choice of the solder joint metallurgy is a key issue for the reliability of the whole assembly.

The electroless Ni/Au plating has gained a high interest for flip chip technology because of its high potential for cost reduction. The process provides a selective autocatalytic metal deposition directly on the aluminum pads of wafers, no costly equipment for sputtering, photo lithography and electroplating is required. Combined with the stencil printing of solder paste it is available for mass production. The usually used eutectic lead-tin solder (PbSn63) on electroless Ni bumps shows a excellent reliability. The suitability of the PbSn63 is limit due to the low melting point of 183 °C. Other solder pastes like PbSn5 and environmental friendly lead-free solders such as Sn-Ag or Sn-Cu are available. However the reliability of such metallurgical systems are not clearly defined for temperatures above 200 °C.

Using the electroless nickel bumping process developed at the Fraunhofer IZM/TU Berlin the plated layers consist of Ni and phosphorous with a phosphorous content of about 9 wt.-%. A small variation of about 1 wt.-% can be measured. The high phosphorous content leads to an amorphous structure of the bump. During the heat treatment up to 250 °C a relaxation of the structure takes place. After a further increasing of the temperature up to 350 °C a crystallization of Ni and Ni<sub>3</sub>P can be observed. The physical, chemical and electrical properties of the Ni bump are also changed, for example the thermal expansion coefficient and the magnetic behavior. This changes of microstructure and of properties may have an influence on the reliability of Nickel bumps. In the paper the results of the influence of temperature on the reliability of the Ni bumps will be discussed. Different reflow tests were carried out and the microstructure of the Nickel bumps were observed with x-ray diffraction measurements and thermal analysis. Especially for the reflow of high temperature solders an impact could be observed.

Beside the advantage for cost reduction the electroless Ni bump shows another proper effect. For the phase formation and phase growth the phosphorous in the bump seems

to inhibit the nickel diffusion into the solder. During the reaction of Ni with tin-based solders the phase  $\text{Ni}_3\text{Sn}_4$  will be formed. A phosphorous-rich layer between the electroless Ni bump and the intermetallic phase  $\text{Ni}_3\text{Sn}_4$  can be observe which reacts as a diffusion barrier. The influence of the amorphous microstructure with its absence of grain boundaries on the phase growth of  $\text{Ni}_3\text{Sn}_4$  phase will be discussed. Results of phase formations and phase growth of lead-tin-solder on electroless Ni will be presented.

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## Integration of micro-mechatronics in automotive applications

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Micro-Mechatronics & System Integration is a multidisciplinary field, which offers low cost system solutions based on the principle of homogenizing system components and consequent elimination of at least one material component or packaging level from the system. These system approaches show, compared to the existing solutions, a higher functionality, more intelligence and better reliability performance. The number of interconnects necessary to link a motor, or sensor, or an actuator to the digital bus decreases, as the smartness of the devices increases.

The paper presents system solutions and manufacturing technology for mechatronic systems, developed at Fraunhofer IZM. To reduce package volume, advanced packaging technologies as Flip Chip and CSP are used, for increased reliability and additional mechanical functionality encapsulation processes as transfer-molding, a combination of transfer- and injection molding or modular packaging toolkit based on LTCC are selected.

The first system is a multi-chip-module for motor control used for automotive applications as window-lifts or sun-roofs. For the module at least three interconnection layers were eliminated using novel concepts as intelligent leadframes and integrated plugs. The package resists harsh environment as present in automotive applications.

Another mechatronic package called TB-BGA or StackPac resp., is a 3-D solution, containing a pressure sensor and the complete electronics necessary for control and data transfers. This package involves CSP-technologies on flex, with an increase of functionality per volume unit by direct CSP-to CSP mounting, eliminating substrate layers and large signal distances of unamplified signals from sensor output.

The mechatronic packages will be discussed in detail. Especially cost, reliability performance and according »Design for Reliability« show the potential of micro-mechatronic solutions in automotive and industrial applications.

## Noncontacting laser based techniques for the determination of elastic constants of thin foils

Anwander, M.; Kaindl, G.; Weiss, B.

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For the miniaturisation of electronic parts in the chip industry it is getting more important to determine elastic and plastic properties of the used materials in their practical dimensions. One geometry of frequently used components are thin foils.

For the determination of elastic constants of thin foils usually the tensile test is applied. In this investigation a microtensile testing machine in combination with a laser speckle extensometer to determine strain is used. The Youngs modulus is deduced from loading-unloading curves.

The laser speckle extensometer consists of two laser diodes, which generates speckles on the specimen surface in a variable distance. The speckles are detected by two optical lenses and two CCD cameras. The displacement of the speckles are recorded by these cameras when the specimen is loaded. The calculation of the displacement and strain, respectively is based on a cross correlation algorithm. The strain resolution of the system is about  $2 \cdot 10^{-5}$ .

Furthermore the clamped foils where excited to vibrations. The excitation is performed by an acoustic source. To detect the frequency spectrum a laser beam is directed onto the specimen and reflected to a differential photodiode. The signal of the photodiode is proportional to the amplitude of the vibrations of the specimen. This signal is processed by a network analyzer in the range between 5 Hz to several 100 kHz. It is well known that the frequency spectrum is influenced by specimen geometry, density, elastic constants and applied stress. Therefore it is feasible to determine Poison ratio and average thickness of the foil by minimizing the differences between the measured and calculated resonance frequencies. Thus the advantages of combining these two measuring systems it is possible to calculate further elastic parameters.

This special setup allows also to monitor in-situ the influence of plastic deformation on changes of the elastic parameters of thin foils.

The applicability of these measurement techniques is demonstrated for several metallic foils with varying thicknesses. Results are presented for rolled and electrodeposited Copper and Nickel foils with thicknesses between 5 and 100  $\mu\text{m}$ .



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## Micromaterials: Materials Science in Small Dimensions

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When the dimensions of material components are scaled down into the micron-range, new effects are found which are only incompletely understood. Alloy formation and microstructure development can be substantially affected because of the short diffusion lengths; synthesis and processing of micro-components are often complicated due to the formation of large internal stresses; and the properties of the material may deviate strongly from those of massive bulk materials of similar chemistry. Especially the mechanical properties in small dimensions are of both academic and practical interest: dislocation plasticity and diffusional creep processes - classical bulk deformation mechanisms - are strongly influenced by the micro-dimensionality; and as means of stress relaxation these mechanisms can greatly affect the operation and reliability of micro-systems.

In this talk an overview will be given of what must be expected when metallic materials are used in the micro-range (»micro-materials«). Emphasis is placed on recent mechanical experiments using several test techniques, ranging from thermal straining (wafer curvature) to the application of external loads in an X-ray diffractometer (microtensile tester), on Cu thin films. The success of different modeling and simulation strategies is critically evaluated. The focus is then placed on the mechanical behavior of Cu and Cu-Al films at high temperatures, where creep and creep damage processes have for the first time been observed. The experimental results are compared with a recent model for diffusional flow constrained by a hard substrate. The results show encouraging, but not complete, agreement. Subtle interface effects not present in bulk materials (e.g. the presence or absence of natural oxide layers) seem to control the damage processes.

To illustrate the importance of interfacial effects in the mechanics of micro-materials, recent in-situ observations by transmission electron microscopy will be shown which illustrate the behavior of lattice dislocations near film/substrate interfaces. Also, for the first time, fatigue processes have been studied systematically in thin film systems; first results show that fatigue must also be viewed as a size-dependent phenomenon - with possible consequences for the design of micro-systems that are still unclear. Overall it is argued that the understanding of size effects coupled with microstructural and alloying effects is indispensable in the rational design of reliable multi-component thin-film systems.

## Nano-scale cutting of single crystal silicon by using friction force microscope and identification of affected layer

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Cutting technique is good for making 3-dimensional structure with high efficiency and also can remove a material without changing the character of the bulk. Additionally, when the cutting depth becomes below the submicron, ductile mode cutting of a brittle material becomes possible. Nano-scale cutting would be a new technique to make complex microstructures. However, it is difficult to control the cutting depth in nano-meter order by the mechanism of a conventional cutting machine. Then, we have developed a nano-scale machining system utilizing the mechanism of a friction force microscope. The system has a stiff cantilever tool, which has a diamond grain on its end as a cutting edge. The cutting depth can be controlled in nano-meter order by giving the constant load onto the cutting edge. The system can demonstrate nano-scale machining of several hard materials, such as single crystal silicon, metals and glasses. In this paper, the character and the identification of the affected layer of single crystal silicon are described. When the  $\langle 100 \rangle$  surface of single crystal silicon is etched by KOH after the  $15\ \mu\text{m} \times 15\ \mu\text{m}$  area nano-machining, machined area remains and gets higher than the other area. This means that the affected layer has the character as the etching mask for KOH. The crystal structure of the affected layer was analyzed by the Rutherford backscattering and the Auger electron spectroscopy. The appearance mechanism of the etching mask effect was discussed. As the result, it is considered that the speed of an anisotropic etching has changed because the affected layer becomes amorphous-like structure by machining.

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## Grating and Moiré methods for micro-measurement

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Grating and moiré methods are revisited with an aim to extending the technique for micro-measurement. Grid methods are widely used in mechanics and engineering for displacement/strain measurement. One of the factors to be considered is the spatial resolution of these techniques, apart from the sensitivity. If the grid lines are assumed to carry the same spatial information as that of fringe line of moiré pattern, grid techniques provides more spatial information than that of moiré, since the grid line frequency is higher than the moiré fringe density. This is the main advantage of grid technique.

In this paper grid techniques are combined with microscopy for micro deformation measurement. High-density grids of 1200 lines/mm are transferred to the specimen surface. The grid pattern is magnified and digitally recorded with optical microscope (OM), scanning electronic microscope (SEM), and atomic force microscope (AFM) respectively. The recorded grid patterns are processed with both 2-D Fourier transformation technique, and sub-pixel techniques for determine the grid spot centroid motion. Thus, the displacement and strain fields are obtained. The spatial resolution and sensitivity for displacement/strain is analyzed both for Fourier transformation method and sub-pixel method. A comparison on the effect of three microscopy techniques is also carried out.

At the same time the unique combination of high sensitivity, optical contrast, range and good spatial resolution, has resulted in Moiré Interferometry being increasingly been employed in the analysis of deformation in electronic packaging. However, for commercial moiré interferometers, spatial resolution of the system leaves much to be desired. The inability of these moiré interferometer to monitor local deformation at the die attachment level or the die corner limits their range of application. This paper presents the development of a fiber optic micro-moiré interferometer, which is capable of monitoring local deformation at micron level. Apart from microelectronics, the system can also be used in applications in micro-mechanics of composite materials, residual stress analysis and bio-mechanics.

Finally, a new strain sensor based on high frequency gratings and two Position Sensor Detectors (PSDs) will be proposed. The high frequency diffraction grating is attached to the surface of a specimen and illuminated by a collimated laser beam. The centroids of diffracted beam spots from the grating are automatically determined with two PSD sensors connected to a personal computer. The shift of diffracted beam spots due to the specimen deformation is then detected. Strain sensitivity of 1 micro-strain can be obtained. The spatial resolution for strain measurement of 0.4 mm is attainable. The system can be used for both static and dynamic test.

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## Mixed mode interfacial fracture toughness investigations for thermo-mechanical reliability enhancement of plastic packages

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Electronic packages like chip scale packages and flip chip assemblies from mechanical point of view are typically rakish compounds of silicon, polymeric materials, ceramics and metals. While ceramics and silicon show a mainly elastic behavior, metals and polymerics show a plastic, viscoelastic and/or viscoplastic behavior which is often highly depend on the temperature. Additionally, polymeric material come with a glass transition temperature which is often inside the temperature range of interest. Unfortunately, the most damaging thermo-mechanical characteristic of polymerics is the much higher thermal expansion coefficients compared to that of silicon, ceramics or metals. These special qualities in combination with various kinds of inhomogeneities and shape owed stress singularities lead to interface delaminations, chip cracking and fatigue of solder joints and in contrast to the goal of the often complex structure, to a thermo-mechanical reliability lower than expected. While life time estimations due to the thermal fatigue of solder joints (typically based on Coffin-Manson type equations) become more and more reliable, the evaluation of the fracture behavior of different polymer/polymer, polymer/metal, polymer/ceramics or metal/ceramics (e.g.) interfaces is of a much lower quality. Therefore, this is an obstacle for comprehensive thermo-mechanical reliability investigations of electronic packages today.

Consequently, the contribution shows the use of non-linear finite element simulations with respect to the non-linear, temperature and rate dependend behavior of different materials used, the application of fracture mechanics concepts (energy release rate, integral fracture approaches, mode-mixity examinations) in combination with experimental investigations. For these purpose, bending specimens consisting of several materials interfaces widely used in FC assemblies have been investigated in particular. In order to evaluate the different approaches used some results have been compared to micrographs from growing interface delaminations by using micro deformation measurements on the basis of a gray scale correlation method applied to micrographs, in particular.

Both, numerical and experimental investigations give the basis for understanding delamination mechanisms in various material interfaces and should support further applications for arising the thermo-mechanical reliability of advanced electronic packages.

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## X-Ray diffraction at elevated temperatures in microsystem technology

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### Introduction

X-ray diffraction is mostly performed at room temperature. However, modern technology increasingly demands new materials usable under extreme conditions, in many cases at elevated temperatures. X-ray diffraction at elevated temperatures complements conventional thermal analytical techniques such as differential scanning calorimetry, thermogravimetric analysis and thermal mechanical analysis. Phase identification, texture analysis or structure investigations are capabilities provided by this method.

### Methodology

X-ray diffraction analysis is a means of identifying a crystalline structure. It is based on the BRAGG equation, which states that X-rays will be diffracted from a crystalline specimen when the following geometrical relationship is satisfied:

$$n\lambda = 2d\sin\Theta$$

where  $n$  is a whole number,  $\lambda$  is the wavelength,  $d$  the lattice spacing and  $\Theta$  is the incident and reflection angle between the X-ray beam and the crystal planes.

If a monochromatic X-ray beam irradiates a flat specimen, and the specimen is rotated through an angle  $\Theta$ , with a linked collimated X-ray detector being simultaneously rotated through an angle  $2\Theta$ , the peaks will be observed at certain angles as the angle  $\Theta$  is scanned. Each peak will correspond to a particular lattice spacing of one phase. The identification of the phase composition of the specimen is achieved by comparing the diffraction pattern with an internationally recognised database. Other materials properties were analysed for instance by the position, intensity or half value breadth of the diffraction peak.

### Instrumentation

At our institute we have at our disposal the »X'Pert-Diffraction-System« and the »High Temperature Chamber HTK 1200«. The temperature in the heating chamber ranges from 25 °C to 1200 °C. The diffraction angle of incidence from  $2\Theta = 0^\circ$ - $164^\circ$  and different geometrical specimens are possible.

### Applications

Different investigations were conducted. First, e.g. the phase transformation of electroless deposited Ni-P at the silicon wafer was in situ analysed. The result of this analysis was that, when a temperature of 300 °C was nearly reached the X-ray amorphous Ni-P-layer changed its phase to Ni+NiP<sub>3</sub>. Second, the phase transformations from ferro-

magnetics Fe-Ta-N-Cr-layers were observed over a temperature range from 400 °C to 600 °C. The knowledge about the formation of various iron nitrids is important for magnetic properties. Third, we have measured structure parameters at various temperatures, after different tempering times at small sensor structures. The half-value-breadth of the diffraction peaks are connected with micro hardness of the layers. Over and above that, taking the measurements of phase specific thermal coefficients of expansion has been included in a near future project.

## Wear resistant Ni-W-B layers deposited by electroplating

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Nanocrystalline Ni-W-B layers containing 58 wt% Ni, 42 wt% W and less than 0.1 at% B were electrochemically deposited and heat treated at 473 K, 673 K, 873 K and 1073 K. Nanoindentation hardness measurements, x-ray diffraction, texture studies and tribology tests have been performed in the as-plated state and following each heat treatment procedure. A possible application as a wear resistant coating is discussed.

## NDE for materials characterization in aeronautic and space applications

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In this paper, nondestructive evaluation (NDE) approaches used at the NASA Glenn Research Center for materials characterization of advanced material systems are described. The emphasis is on high-temperature aerospace propulsion applications. The material systems include monolithic ceramics, superalloys, and high temperature polymer, metal, and ceramic matrix composites. In the aeronautic area, examples of cooled ceramic plate structures, gamma-TiAl blade materials for low-pressure turbines, metal matrix composite rings for compressor disks/blisks, and ceramic matrix composite blades for high-pressure turbines are presented. In the space area, examples of cooled carbon-ceramic composite for gas generator combustors and polymer matrix composite for flywheel energy storage at the space station are presented. Manufacturing problems, effect of defect results, and the use of NDE-based finite element modeling are also highlighted. NDE technology needs for improved nondestructive characterization of micro materials and components are identified and discussed.



## The formation of the $\text{Dy}_x\text{O}_y$ films on the InP substrates

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In the last years there has been much interest in metal-insulator-InP field-effect transistors. The study of new materials as a gate isolators in MIS structures based on InP substrates is important because charge properties of the insulator-InP interface are still not good as those of the thermally grown  $\text{SiO}_2$ -Si interface. Our preliminary results obtained in the Si and InP MIS structures with the dysprosium oxide ( $\text{Dy}_x\text{O}_y$ ) films gave possibility to suggest such films for investigation as a gate insulator in the InP MIS structures.

The  $\text{Dy}_x\text{O}_y$  films with the thickness  $d_{\text{ox}} = 30.0\text{--}70.0$  nm were prepared by evaporation dysprosium Dy: (1) in the vacuum ( $3 \times 10^{-6}$  mm Hg), or (2) in the  $\text{O}_2$  environment ( $3.5 \times 10^{-5}\text{--}1.2 \times 10^{-3}$  mm Hg), and with subsequent oxidation of Dy films on n-InP (100) substrates in dry oxygen stream (0.1–2 litre/min) at the temperature  $T_{\text{ox}} = 300\text{--}350$  °C for time  $t_{\text{ox}} = 5\text{--}30$  min. The charge properties of Al- $\text{Dy}_x\text{O}_y$ -n-InP (100) structures were studied by means of 1 MHz C-V method.

It is shown that the properties of the  $\text{Dy}_x\text{O}_y$  films and  $\text{Dy}_x\text{O}_y$ -InP interface to a great extents depend on  $T_{\text{ox}}$ .

At the  $T_{\text{ox}} = 300\text{--}320$  °C the  $\text{Dy}_x\text{O}_y$  dielectric films are formed at the initial stage of the oxidation process. The  $\text{Dy}_x\text{O}_y$ -InP interface has positive effective charge  $Q_{\text{ss}}$ , the injection-type hysteresis 1–2 V and breakdown field strength  $E_{\text{BR}} = 10^5\text{--}10^6$  V/cm. As the  $t_{\text{ox}}$  and/or  $\text{O}_2$  stream increase, oxygen atoms diffuse through the  $\text{Dy}_x\text{O}_y$  film and InP native oxide film begins to grow. The charge properties of the MIS structures depend on the properties of the InP native oxide film-InP substrate interface. To avoid the InP surface oxidation it is necessary to passivate it chemically, for example, in the  $(\text{NH}_4)_2\text{S}_x$  water solution.

At the  $T_{\text{ox}} > 320$  °C the interaction between  $\text{Dy}_x\text{O}_y$  film and InP substrate takes place. As a result, the transition layer consisting of  $\text{Dy}_x\text{O}_y$  and  $\text{P}_2\text{O}_5$  begins to form. As the  $t_{\text{ox}}$  and/or  $\text{O}_2$  stream increase the transition layer thickness increases too. In this case MIS structures with the  $d_{\text{ox}}$  about 70 nm obtained by Dy evaporation in the vacuum (1) and in the  $\text{O}_2$  (2) have the following properties:

(1)  $E_{\text{BR}} \sim 5 \times 10^6$  V/cm; current density  $j \sim 3 \times 10^{-8}$  A/cm<sup>2</sup> at the 10 V;  $Q_{\text{ss}} \sim \pm 1 \times 10^{11}$  cm<sup>-2</sup>;  $N_{\text{IT}} \sim 1 \times 10^{11}$  cm<sup>-2</sup>eV<sup>-1</sup>; ionic-type hysteresis 1–3 V.

(2)  $E_{\text{BR}} \sim 2.5 \times 10^6$  V/cm;  $j \sim 0.94 \times 10^{-3}$  A/cm<sup>2</sup> at the 10 V; positive  $Q_{\text{ss}} \sim 3 \times 10^{11}$  cm<sup>-2</sup>; surface state density  $N_{\text{IT}} \sim 5 \times 10^{11}$  cm<sup>-2</sup>eV<sup>-1</sup>; injection-type hysteresis < 0.3 V.

There is the reason to make conclusion that the method of the thermal evaporation of Dy in vacuum or  $\text{O}_2$  environment with subsequent oxidation of Dy films gives the possibility to obtain  $\text{Dy}_x\text{O}_y$  films and  $\text{Dy}_x\text{O}_y$ -InP interface with rather good properties. This method is interesting in the further investigation for deposition of  $\text{Dy}_x\text{O}_y$  films as a gate insulator in the InP MIS structures.

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## Contactless current detection at megahertz band width via the implementation of the heterodyne mixing technique

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A development of a non-destructive and contactless test method for measuring circuit-internal currents is one of the great demands of the function and failure analysis engineering. As the recent development trend of ICs to a permanent reduction of the power dissipation and to the rise of the operating frequency shows, such an internal test method has to offer an excellent sensitivity and a wide band width. Already, our group proposed anisotropic magneto-resistive (AMR) and giant magneto-resistive sensors for the internal current detection via evaluation of the magnetic field caused by a current carrying conducting line. We demonstrated also its ability to detect currents down to the  $\mu\text{A}$ -range and to achieve quantitative current determination with kilohertz band width [1][2]. Managing a higher band width is difficult because frequency dependent parasitic signals arising from the current carrying conducting line disturb measurements. In this paper we therefore introduce a test method for current detection with megahertz band width via the implementation of the heterodyne mixing technique.

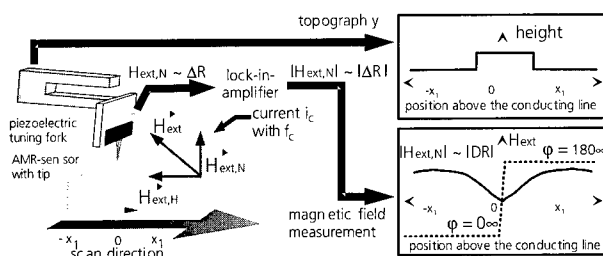


Figure 1 : Measurement principle and setup for topography measurement and current detection via evaluation of magnetic fields

For this work, a new bifunctional probe consisting of an AMR-sensor for current detection and of a sharp tip for topography measurement is used (Fig. 1). In the conventional measurement setup, the AMR-sensor is usually supplied with a dc-voltage. Its magnetic field sensitive resistivity change  $\Delta R$ , caused only by the normal component  $H_{ext,N}$  of the magnetic field  $\vec{H}_{ext}$  occurred from the current  $i_c$ , is measured as an absolute magnitude  $|\Delta R|$  and a phase  $\varphi$ . A linescan of  $|\Delta R|$  and  $\varphi$  perpendicular to the current carrying conducting line shows a  $|\Delta R|$  of zero and a  $180^\circ$  phase shift at the middle of the conducting line.

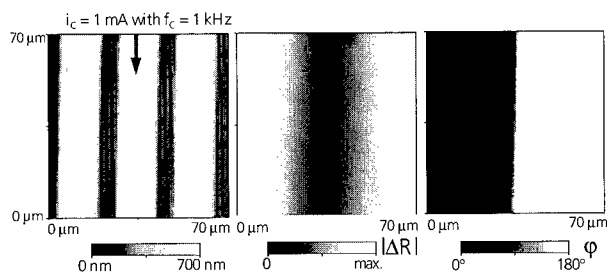


Figure 2 : Measured topography image and areascan of  $|\Delta R|$  and  $\varphi$  as the result of a current detection of 1 mA with  $f_c = 1 \text{ kHz}$

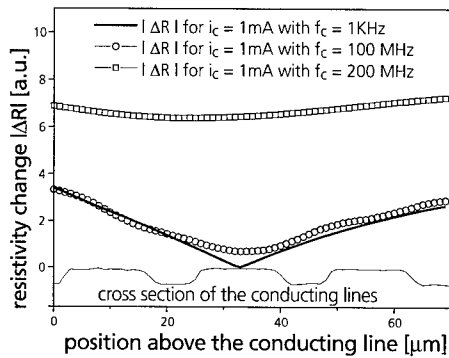


Figure 3: Investigation of the frequency limit of the current detection by using a conventional method

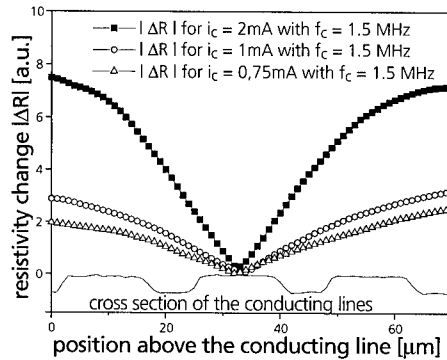


Figure 4: Current detection at  $f_c = 1.5$  MHz by using the heterodyne mixing technique

This conventional measurement setup shows a frequency limit of 100 kHz due to the superimposed parasitic signals. In order to eliminate these parasitic signals the heterodyne mixing technique is applied. At this new measurement setup the power supply to the AMR-sensor is now an ac-voltage with a frequency  $f_s$ . Due to the heterodyne mixing technique signals with sum frequency  $f_s + f_c$  and difference frequency  $f_s - f_c$  appear. These signals are proportional to the magnetic field to be measured. Therefore, only the signal component with the difference frequency  $f_s - f_c$  is measured. Because the frequency component  $f_s - f_c$  lies in the low frequency range, this signal can be easily processed further on.

All measurements were performed on three 16  $\mu\text{m}$  wide parallel conducting lines. Only, the middle conducting line was fed with a current  $i_c$  of 1 mA with  $f_c = 1$  kHz. At first, the topography image and the areascan of  $|\Delta R|$  and  $\varphi$  (Fig. 2) were obtained by using the conventional setup. The result clearly presents the functioning of the current detection. Then, the frequency  $f_c$  of the current was increased. The obtained linescans of  $|\Delta R|$  across the conducting lines show that a current detection at  $f_c = 220$  kHz is not possible (Fig. 3). By using the heterodyne mixing technique a current detection up to a frequency  $f_c$  of 1.5 MHz is possible as shown in Fig. 4. A more detailed discussion will be in the full paper.

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## Strength analysis of a micromechanical acceleration sensor by fracture mechanical approaches

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Today, the production of acceleration sensors for automotive airbag systems is based increasingly on Si micromechanics. Fig. 1 shows the schematic functional principle of a sensor fabricated from single crystalline Silicon by bulk micromachining technologies. The key element of the sensor consists of a seismic mass element which is suspended on a frame. The acceleration of the sensor leads to a displacement of the mass resulting in a bending deformation of the suspension element. The induced bending strain can be recorded by piezoresistive films providing an electrical signal which is related to the acceleration intensity.

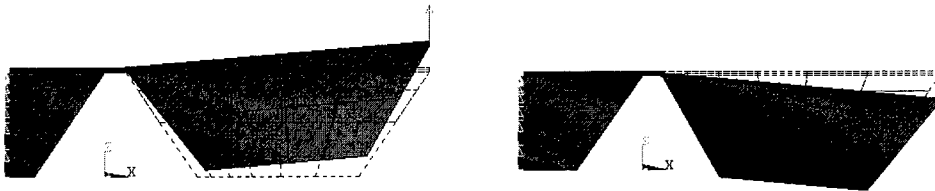


Figure 1: Finite Element model of the loaded acceleration sensor element

To ensure a reliable function of the system during application, the sensor design has to consider the strength properties of the moving sensor component. 3D Finite Element Model (FEM) simulations (Fig. 1) were performed to analyse the deformation behaviour. In addition, an indentation testing apparatus was applied for experimental bending strength tests. The investigations have shown that the strength of the system is mainly controlled by local stress concentrations occurring at different spots of the suspension element during bending. If the seismic mass element moves upwards, a local stress concentration occurs at the notches induced by anisotropic wet etching at the lower side. If the seismic mass elements is bent downwards, local stress concentrations occur in the patterned top surface of the suspension due to edges and different elastic properties of passivation films and Si. As a consequence, different appropriate fracture mechanics approaches, like a notch stress intensity concept and a  $d^*$ -concept, must be applied for a theoretical strength assessment. The analysis has shown that the fracture stress depends distinctly on the layout of the passivation structures in the suspension surface and on the notch geometry. Thus, the investigations can be used for a layout optimisation with respect to strength. Furthermore, the maximum local stresses during application could be shown to be less than 15% compared to the fracture limit for both bending directions. As a consequence, a high mechanical reliability of the sensor type during application can be expected.

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## Lifetime investigations of directly wafer-bonded samples under static and cyclic loading

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Wafer bonding is one of the key technologies in the fabrication of micromechanical components, like pressure sensors, acceleration sensors, angular rate sensors or micropumps. During use, these bonded devices are exposed to continuously or periodically acting mechanical stresses, particularly due to vibrations, thermo-mechanical mismatch or internal or external pressures. The aim of this study is to investigate the strength behavior of mechanically long-term loaded wafer-bonded interfaces. The knowledge of these properties is of considerable significance with respect to the mechanical reliability of micromechanical sensors and actuators during industrial application.

Strength and fatigue tests of pre-cracked fracture mechanical samples were performed to investigate the lifetime properties of wafer-bonded samples exposed to constant (static) or cyclic stresses as a function of loading intensity. The investigations included a study of the influence of different bonding conditions, like hydrophilic, hydrophobic and ultra high vacuum conditions, on mechanical reliability properties.

All hydrophilic samples loaded at stress levels above of 40% of the initial (short time) strength failed if the loading duration was extended up to one year (Fig. 1). A similar behavior was found for different bonding treatment conditions as well as for the cyclic loading experiments. However, the intensity of fatigue depended on the treatment conditions and the loading type parameters.

A fracture mechanics approach was applied to develop a theoretical model of lifetime properties. The approach is based on experimentally measured data of the subcritical growth of the pre-existing cracks. The theoretical prediction agrees with the experimental results, both static and cyclic loading experiments, very well (Fig. 1). Therefore, the concept proposed can be applied for a reliability assessment of wafer-bonded micromechanical components.

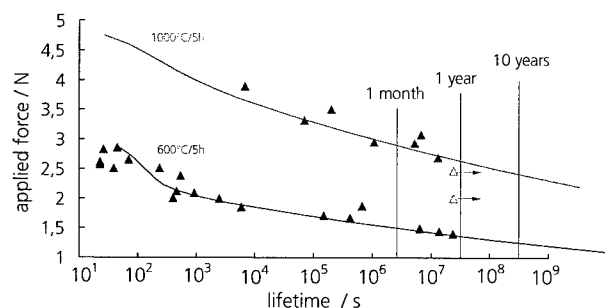


Figure 1:  
Results of experimental static  
lifetime investigations (symbols)  
and theoretical prediction (line) of  
differently annealed samples.

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## Environmentally assistant damage of metals with covering

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As well known, in conditions of hydrogen embrittlement, the hydrogen renders reduced fracture toughness of many metals and steels affecting behaviour of engineering structures. The hydrogen absorbed by a metal is usually dissolved in the lattice in the proton (atomic) form. A part of protons reaches the surface of pre-existing or freshly created cracks where they recombine with electrons and form molecular hydrogen in the crack cavity. Because effective radius of hydrogen molecules usually exceeds the size of vacancies in the lattice cell, the molecular form of hydrogen is thermodynamically more stable near the crack surfaces which leads to the accumulation of hydrogen inside the crack. The appearing excessive pressure can be especially dangerous when the metal surface is coated and small delaminations appear more frequently. Fracture in conditions of hydrogen embrittlement takes place even in the absence of any external loading, that is, only under the excessive pressure of hydrogen accumulated inside the crack.

In this paper, a theoretical model of quasistatic growth of environmentally assistant delaminations is proposed. For the case of a penny-shaped delamination the main equations describing its growth is derived. The expressions for incubation time and the closed-form solutions of the equation are obtained. It is shown that after incubation time, the contour of a delamination begins to spread with the finite velocity, which doesn't change in process of growth. Phenomena of slow kinetic propagation of hydrogen assistant cracks in metals is known and observed in many experiments.

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## A method of transfer relations – new perspectives in physical grounds of near-field optics

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As a consequence of the first principles of the wave multiple scattering theory, a new non-perturbative approach, so called transfer relations method [1], is applied to wave scattering from a periodic interface  $z=f(x)$  of two dielectric media in the rectangular coordinate system  $x,y,z$ . According to this approach, a transitional region between media is subdivided into a stack of  $N$  elementary layers perpendicular to the  $z$  axis and separated from one to another by an infinitesimal splits. Transfer relations are derived in the form of a system of exact matrix equations for the matrix wave reflection and transmission coefficients of the stack of  $N$  layers and the matrix wave amplitudes (local fields) of waves in splits between layers. The transfer relations give a differential basic Riccati equation for the matrix wave reflection coefficient from and a corresponding differential equation for the matrix wave transmission coefficient through the periodic interface, as well as a differential equation for a local field inside the transitional region, the embedding parameter of these differential equations with given initial or »final« conditions is taken along the  $z$  axis.

The derived basic Riccati equation is solved numerically, using the Bloch-like mode representation and supposing the electric wave field being parallel to the  $y$  axis. It is shown, in particular, that well known Wood's anomalies of the wave reflection field in the near-field zone of the interface are related to the mutual resonant transformation between propagating and evanescent Bloch-like modes by a Bragg-like wave diffraction on the periodic interface. An asymmetry property of the Wood's anomalies is pointed out. What is more, an asymptotic solution to the Riccati equation obtained in the case of large dielectric permittivity of the irradiated medium and the two-mode approximation predicts a logarithmic singularity in the Wood's anomalies.

At last, the problem of the mapping of the Wood's anomalies in the near-field of the interface into the far-field of the probe grating is considered with the aid of the transfer relations approach. A possibility to obtain from this mapping an imaging of the interface parameters is discussed.

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## High performance thermosets – an overview

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The rapid growth in manufacturing and applying high-performance microsystems generates increasing demand upon thermosets with advanced properties. To meet the requirements for applications like

- conductive adhesives for packaging of advanced microprocessor chips,
- encapsulation materials and protective coatings for semiconductor devices,
- binders for laminates and thin-film insulation for high-density, high-speed multilayer circuit boards and multichip modules,
- thin layers for optical wave guides,
- matrix-materials for non-linear optical devices

and others, there is a need for materials with unique combinations of properties. This can not be often realized by common thermosets. The main requirements are focused on high thermal stability above 220 °C, high glass temperature above 220 °C combined with high toughness, high resistance against water, low electrical dissipation factor low inherent flammability and low corrosion potential.

A comparison of the potential of the different types of thermosets with excellent thermomechanical properties will be drawn.

**Keywords:** high performance thermosets



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## An analysis of crack nucleation in fibre-reinforced concrete

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An improvement of ductility in high strength concrete can be reached by adding short steel fibres. This is the case of Reactive Powder Concrete (RPC) developed on the beginning of the nineteen's. This material is made of a very fine homogeneous and compact matrix and short steel fibres. Fibres have been added to concrete to create pseudo-strain-hardening. The bridging fibre action can be very beneficial to increase the stress carried across crack faces. In order to optimise the mechanical behaviour (including crack initiation and damage process) of this new generation of concrete, the influence of the orientation of fibres due to the process of casting must be studied.

Crack nucleation is investigated theoretically in terms of stress concentration due to the elastic anisotropy of a locally oriented mass of fibres embedded in a compact matrix. By considering a locally oriented fibres volume as an inclusion in a homogeneous medium, the Eshelby's inclusion model provides a description of the microstructural stress concentration and a model for crack initiation based on stress intensity factor in mode I is developed. Numerical results show that cracks can be nucleated under tensile stresses at the inclusion-matrix interface.

Specimens of RPC reinforced by oriented fibres have been tested under uniaxial tensile stress. The analysis of s-e curves and samples, after each test, have shown the influence of the oriented fibres on the beginning of the damage process. These observations support the model of crack nucleation based on the Eshelby's inclusion.

## BGA packaging technology for rapid prototyping supported by advanced analytics

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A main focus of Fraunhofer IZM's mechatronic and Encapsulation Technology Group is the Development and Manufacturing of microelectronic packages for a large variety of applications, including highly reliable mechatronic packages for automotive and industrial applications.

Within an internal research program, the Analytic Pool, various analysis techniques have been applied to increase the knowledge on package failure mechanisms and critical designs and to yield maximum reliability within new package development. For the investigations described in this paper the BGA package type has been selected.

This geometry offers optimum properties for the rapid prototyping of Single and Multi Chip Modules and allows cost effective evaluation of new technological approaches.

The analytic tools applied covered the fields of material analysis, e.g. IR spectroscopy, of nondestructive testing as X-ray and Acoustic microscopy and of destructive analysis as cross-sectioning and subsequent SEM analysis.

Conclusions gained from the investigations performed are summarized in package design rules for microelectronic packages and an outlook on application for advanced packages as mechatronic packages is given.

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## Application of electroplating in MEMS-micromachining exemplified by a microrelay

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Micro-electro-mechanical-systems (MEMS) are increasingly employed in industrial sensor and actuator applications. Even in the age of semiconductor power switches, mechanical relays have not yet become dispensable in technical applications due to EMC-aspects, mechanical separation of current lines or electrical isolation of control and load circuit. Automotive and communication sectors of industry demand a high number of mechanical relays and therefore provide a promising market for electrical contacts developed with microsystem technologies (MST). MEMS-relays could be favoured for size reduction, SMT-capability, integration with microelectronics and improved dynamic behaviour and reliability.

Actuation principles and fabrication in MST normally differ from traditional relay technologies based on current driven electromagnetic coils. The suitability of electroplating for the processing sequence of an electrostatically actuated MEMS-relay, assigned for current loads of several amperes, will be illustrated. Thereby, the maximum current load depends on actuator features like displacement or contact gap, switching dynamics or contact force as well as on material properties like hardness and elasticity. The system elements of the microrelay are fabricated in surface micromachining (SMM) dominated by micro-electroplating. Depending on the intended use as passive contact material, coating, solder alloy, functional or sacrificial layer, different sorts of metals and electroplating procedures are employed.

The electroplated contact system consists of two fixed contacts and a movable shorting bar of Ni. Coating alloys like AuCo, AuNi or PdNi, electroplated on the surface of the contact system, influence switching properties like contact resistance, electro-mechanical abrasion and sticking effects. The shorting bar is connected to electroplated cantilever beams of Ni or NiFe, which bend out of plane due to residual stress and which can be actuated by electrostatic or magnetic force. Electroplated Ni or NiFe exhibit favourable mechanical properties. The electroplating procedure can be modified by timing or bath compounds to influence mechanical stress factors in order to bend the beam out of plane.

As electrostatic actuation in MEMS requires small airgaps, the sacrificial layer for the movable elements is electroplated of copper with less than 1  $\mu\text{m}$  layer thickness. The modified procedure of patterned deposition without plating mould ensures good uniformity and smooth edges, which is elementary for the electrostatic pull-in.

The establishment of contact force depends of the stiffness and overtravel of the contact spring. The use of electroplating allows to deposit the fixed contacts after removing the sacrificial layer, effecting that the overtravel can be defined by the height of the fixed contacts.

The device is intended to be encapsulated by bonding a bulk-micromachined wafer onto the systemwafer using electroplated solder alloys. In order to keep the line resistance low, the device wafer is bulk-micromachined from the backside and filled up by electroplating. Finally, the encapsulated chip can be put onto a board by reflow soldering resulting in a device height on board of about 1mm.

## Voltage contrast measurements with an electric force microscope based test system

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The complexity of present integrated circuits (ICs) requires a contactless chip-internal test technique for function and failure analysis. The trend towards increasing working frequencies and decreasing structure widths of ICs pushes the present standard test technique for contactless chip-internal measurements, the electron beam test (EBT) [1], to its limits concerning spatial and time resolution. The spatial resolution of EBT is about  $0.7\text{ }\mu\text{m}$  and the time resolution about 50 ps [2]. Thus, the EBT cannot follow the requirements of future IC generations. A new test technique, the electric force microscopy (EFM) [3] seems to be able to solve the problems of EBT. So measurements at structures down to a width of 75 nm [4] could as well be shown as measurements with working frequencies up to 110 GHz [5].

Scanning force microscopy (SFM) [3] is a powerful tool for surface analysis with high spatial resolution. This measurement technique uses a probe consisting of an atomically-sharp tip fixed to a cantilever, which is moved above the sample. The interesting surface information is measured by detecting the bending of the cantilever generated by a tip to test-point force interaction. The EFM uses this technique to measure electric signals with nanometric spatial resolution. By detecting the Coulomb force interaction (CFI) between a conductive probe-tip and the test-point, it is possible to get information about the electrical potential of the test-point. Because of the conductive probe tip the bending  $\Delta h$  of the probe is caused by the voltage difference between the probe and the test point (Fig. 1). To minimize disturbing atomic force interactions between the probe and the sample it is necessary to retract the probe to a constant height  $z_0$  of typically 50 nm from the surface of the device under test (D.U.T.). Two-dimensional measurements of the device voltage amplitude (voltage contrast) can be made by scanning the probe above distinct device areas and by measuring at every point along the scan path (Fig. 3,4). Here, bright areas represent high voltage amplitudes and dark areas low voltage amplitudes.

In the full paper EFM-basics and applications will become explained. An introduction in the heterodyne mixing technique (Fig. 2), which allows to measure the amplitude and the phase of harmonic signals with almost unlimited height of frequency, will be given and EFM-measurements of 2-dimensional voltage distributions will be presented (Fig. 3). Furthermore insights into actual research concerning EFM-measurements at deep-sub-micrometer structures (Fig. 4) will be given.

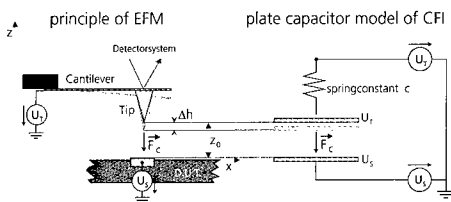


Figure 1: Principle of electrical force microscopy

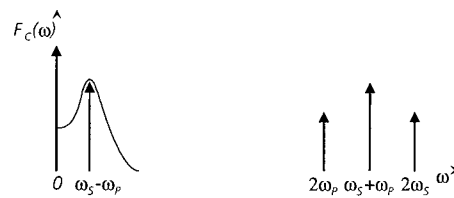


Figure 2: CFI-frequency-spectrum by the use of the heterodyne mixing technique

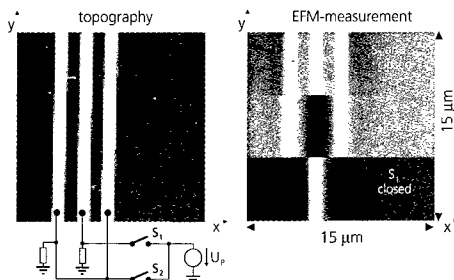


Figure 3: Topography and EFM-voltage contrast image

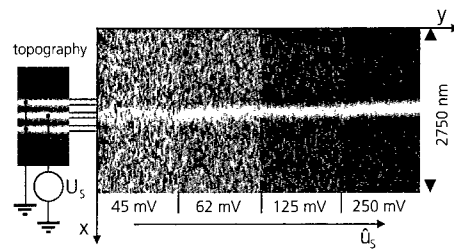


Figure 4: EFM-measurement at 100 nm conducting lines with different voltage amplitudes

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## Migration-resistant amorphous AlY thin film metallizations for SAW devices

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Amorphous metal alloys are ideally suited for interconnects in micro-electromechanical systems (MEMS) because of their resistance against migration and diffusion, and their stability in chemically aggressive environments, which should both lead to a substantial improvement of lifetime and reliability of robust sensors. While amorphous refractory metal alloys and amorphous silicides are excellent interconnect materials for devices operating at elevated temperature, these systems lack the cost-effective and easy interconnect processing of the prevalent polycrystalline aluminium alloy metallizations. Amorphous aluminium alloys are, dependent on their composition, limited to devices operating at a temperature of  $T = 100\text{ }^{\circ}\text{C}$  to  $T = 200\text{ }^{\circ}\text{C}$ , but their stress-migration resistance and chemical stability is quite comparable to amorphous refractory metal alloys and amorphous silicides. Aluminium-rare earth alloys seem very promising for processing amorphous interconnects, in particular because of their high strength and ductility, though having low density, and their relatively low electrical resistivity compared to other amorphous metal alloys. Therefore these metallizations are especially suited for surface acoustic wave (SAW) sensors, where the interconnects are exposed to considerable mechanical strains. In this work amorphous AlY alloy thin film metallizations deposited on single crystal silicon substrates at room temperature (R.T.) by ultra-high vacuum (UHV) electron beam evaporation will be presented, and their mechanical and electrical properties together with their crystallization temperature will be investigated.

**Key words:** Nanomaterials, Amorphous Metals, Thin Films, Sensor Materials

## Deformation and cracking at microindentations in GaAs

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The paper deals with basic mechanical properties of single-crystalline GaAs as revealed by indentation testing. The investigations were performed on (001) surfaces of differently doped GaAs single crystals which were grown by the LEC or VGF techniques, resp. Additionally to the hardness in the micro- and ultramicro-ranges we analysed the asymmetric dislocation mobility, e.g. by tempering and AFM techniques, and the cracking patterns around the indentations. This includes the evaluation of the influence of the loading level on the mode and frequency of cracks emanating from the indentations.

From these experiments we found a critical load for »external« cracking (Fig. 1) of the order of 0.1 N as shown in Fig. 2. Obviously this critical load for the appearance of cracks is determined by dislocation controlled crack initiation rather than by fracture mechanical growth of preexisting defects.

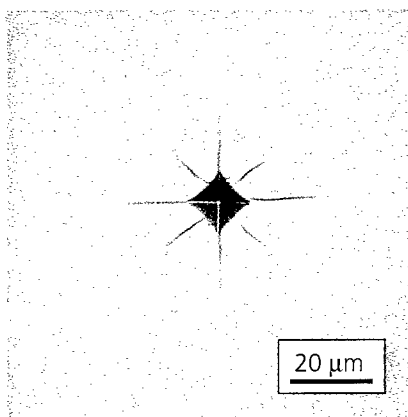


Figure 1:  
Typical cracking pattern around Vickers hardness indentations in GaAs,  $F=0.98\text{ N}$ , (001) surface,  $\langle 110 \rangle$  orientation parallel to the indentation diagonals

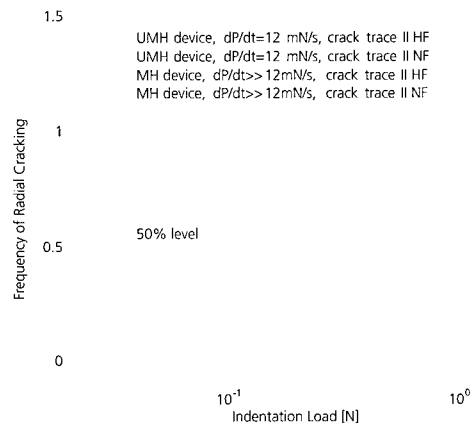


Figure 2:  
Frequency of the counted number of cracks parallel to the indentation diagonals in dependence on the indentation load, C-doped GaAs, UMH=Ultramicro-indentation, MH=Microhardness,  $dP/dt$ =loading rate



## On a deformation of the polycrystalline structure

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The technology of microparts or microcomponents has a special character with regard to the rate between the size of the component and one of the material structure. The polycrystals are materials which are often used in an engineering and so the history of their research is going through a long time.

Their study is connected with discoveries in the field of X-ray and microscopy techniques. While the optical microscopes can show mainly the structure of polycrystals – a system of grains with boundaries, the X-ray, SEM and TEM instruments can show the atomic structure of individual grains and so in the whole polycrystalline system, where grain boundaries are assumed as obstacles for a movement of dislocations only. These two levels of a structure of the polycrystal represent two different models of deformation mechanisms, serving as explanation, for theoretical description of their mechanical properties.

The theories of elasticity and plasticity, which are based on the preposition of homogeneous anisotropic continuum, were a good starting conditions for a description of elastic, but especially of plastic properties of polycrystals at the beginning of sixtieth. The model which was deduced from experiments was named later by Kröner »continuized crystal«. The paper of Iwakuma and Nemat-Nasser [1984], based on the model of the localized deformation, in which the overall moduli of polycrystalline solids are estimated, the rotation of grains takes place, but without presence of grain boundaries. The main advantage of this concept is the possibility to express the constitutive equation of polycrystals by macroscopic strains and stresses.

Many experiments which were carried out with polycrystals during the second half of the century were summarized and generalized by J.F.Bell [1968]. The differences between the calculations which were obtained under preposition that grains don't rotate mutually and Dawson's experiment [1968], where rotation of grain was detected rentgenographically, showed on a discrepancy of the continuous model.

Simultaneously and intensively was investigated in the same time the structure of grain boundaries. Meuris and Hornbogen [1976] showed for age-hardening Al alloys that in the vicinity of grain boundaries exist particle free zones with low yield point, while the interior of grains is much stiff.

The first point of view on the polycrystalline structure, i.e. the system of grains with boundaries, didn't have in sixtieth a theoretical background for a further development. Only individual experimental works were done in these years. The paper of Rovinski and Sinajski [1959] belong between them. They studied rentgenographically the movement of polycrystalline grains on a surface of cylindrical specimen from Al alloy. The obtained diagrams show on a very great differences among the deformation states of individual grains and the stress-strain diagram of the whole specimen. They recorded also reversible springs at the rotation of grains.

We have started our studies at the end of eighties with measurements of elastic moduli of Al polycrystals. Further we have analyzed the rotation of grains by using of micro-stereoimage technique - J.Mater.Sci [1984] – and by photoelastic modeling. The specimens and the models were loaded as statically so dynamically. The discussion of results of these experiments will be introduced in the presented paper. The examples of technologically induced polycrystal deformations will be presented.

## Elastic modules of solid $C_{60}$ : Measurement and relationship with nanostructure

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Elastic moduli of solid  $C_{60}$  have been measured in single crystal of primary fullerite and diverse metastable phases of polymerized and amorphous  $C_{60}$  synthesized under high-pressure high-temperature (HPHT) conditions within the wide range of pressure  $P = 5\text{--}13$  GPa and temperature  $T = 500\text{--}1870$  K. Small sizes and heterogeneity of HPHT samples admit application of the specific microacoustic technique. A focused ultrasonic beam and ultra-short probing pulses have been employed to measure sonic velocities and study elastic module within area  $100\text{--}300$   $\mu\text{m}$  diameter. Ultrasonic measurements have revealed permanent increase of elastic module  $K$  and shear module  $G$  get compared with those in diamond for 3D polymerized  $C_{60}$ . Amorphous  $C_{60}$  produced at the highest pressure  $P = 13$  GPa and temperature  $T = 1600\text{--}1900$  K possess unique mechanical properties including the highest value of the longitudinal sound velocity ( $c_L = 26$  km/s) among substances known now and values of the bulk module ( $K = 700\text{--}800$  GPa) substantially greater than the bulk elastic module in diamond ( $K = 440\text{--}490$  GPa). Essential elastic heterogeneity of the most HPHT states has been shown including smooth distribution of elastic properties over a specimen for crystalline polymerized phases.

Experimental results as well as model calculations developed on the base of the finding evidence that elastic properties correlate with state of fullerene molecules, character and number of covalent bonds and nanostructure of the matter. Measuring elastic properties is one of the effective way to get information on nanostructure of solid  $C_{60}$ , to classify and identify its diverse phases.

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## Implanted pressure sensors on CVD diamond films

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To the best of our knowledge, this work is the first publication on an all in diamond pressure sensor. It is manufactured on a CVD diamond substrate by means of implanted sensitive elements. The piezoeffect of boron doped diamond films is known from earlier literature [1]. In previous works, the doped resistances were deposited and subsequently structured through an in-situ-doping during the CVD process. In this work, ion implantation was used. With this method, the structuring of the sensor elements was considerably simpler. The damages caused by the ion beam during implantation can be removed through an additional temperature process because only a low implantation dose was used. Optical grade CVD diamond substrates were used for the manufacturing of pressure sensors.

The temperature and pressure dependency of the sensors were investigated. The diamonds used for the manufacturing of the sensors had dimensions of  $5 \times 5 \times 0.2 \text{ mm}^3$  and were polished on both sides. The measurement of the photoluminescence spectrum, which delivers the species of the contamination, was used for the preselection of high quality diamond substrates. Nitrogen and silicon were the only detectable defects. However, their concentrations were far below those found in the substrates of thermal grade CVD diamond films.

The implanted sensors were connected to a bridge circuit. The four sensors were arranged on the membrane in such a way that by pairing appropriately, the transversal or longitudinal effect superseded. Because these two effects effectuate an opposite change in resistance, the bridge's voltage increases with increasing pressure.

The manufactured sensors were placed on isolated sensor holder made of steel. The pressure on the membrane was exerted by nitrogen. The pressure range used on the mm diameter round membrane was 0-70 bar.

The bridge signal is sensitive for a pressure range of 20-70 bar.

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## Reflective bistable liquid crystal displays on flexible substrates

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Reflective bistable Liquid Crystal Displays on flexible substrates without polarizers are of increasing interest for handheld and portable applications. This is due to the fact that reflective LCD devices can be operated without backlight leading to a reduction in power consumption as well as a reduction in size and weight of the display. The bistability of a display device offers the opportunity of storing information without energy consumption. Further weight reduction of displays can be retained by the replacement of the glass by a polymer substrate. In addition, polymer substrates are particular interesting due to safety considerations for applications in automotive, avionics, portable electronic consumer products or electronic toys. Being unbreakable is also a prerequisite for the use of displays in smart cards. Other applications for such displays are low cost products such as electronic stickers for use in warehouses, modern offices and parcel services.

Although first reflective displays on flexible substrates have been presented in the past, there is still further demand on material development for both the flexible substrate and the bistable liquid crystal cell. The work presented in this contribution is concerned with both aspects: Improved cholesteric liquid crystal mixtures are introduced in a display exhibiting high reflectivity and mechanical stability and new bistable effects are introduced with the aim to decrease the switching times between different bistable states. Furthermore, the suitability of different polymers as display substrates is discussed.

## Large area surface-relief microstructures for optical applications

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By microstructuring the surface of materials, their optical properties can be modified. Periodic structures in the sub-wavelength scale lead to an effective graded-index profile, which reduces reflection (anti reflection or AR structures). Structures with typical dimensions above the radiation wavelength lead to a deflection of incident radiation. Stochastic super-wavelength structures lead to a reduction of the specular reflection by distribution of the radiation into different directions (anti glare or AG structures). Periodic microstructures in the range of the incident wavelengths lead to resonance phenomena, especially on metal surfaces.

Microstructures like these can be produced by holographic exposure processes on large areas. A masterstructure is generated first in photoresist. Afterwards this photoresist structure can either be used to fabricate a nickel stamper for embossing or as an etching mask. With our setup periodic surface relief structures with grating constants from 200nm up to 60µm with various grating types and profile shapes can be realized with a good homogeneity on an area with a diameter of 45cm.

We used nickel stampers to emboss surface relief microstructures on transparent polymer materials in order to achieve AR and AG surfaces. Various replication processes have been investigated: roller UV embossing, hot embossing and injection molding. The structures can be used for visible applications. In order to optimise reflection performance for display applications, AR and AG structures can even be combined to a multifunctional surface, that can be replicated in a single process. Also AR surfaces for the much broader solar spectrum can be realized by subwavelength gratings. The mentioned production methods have the potential of a cost-effective mass production.

Periodic microstructures on metal surfaces can be produced by etching. The photoresist structure is used as a mask. Different etching technologies have been investigated. Periodically microstructured metal surfaces show a wavelength-selective emissivity. Also thin metal films have been perforated by periodic structures in the µm-range. These act as low absorption bandpass filters for IR radiation. By changing the structure parameters, these radiation management devices can easily be tuned to design wavelengths. We intend to use these devices in thermophotovoltaic energy converters in order to increase their effectivity.

## Micro-metalforming with silicon dies

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### Introduction

The introduction of forming technology to the manufacturing of MEM components makes great demands on the applied dies. The dies should be featured by a high strength and hardness, a low surface roughness, and a good micro-structurability. Silicon is one material meeting all these requirements. Using lithography and etching processes smallest structures with highest precision and surface quality can be produced.

### Experimental results

For micro-metal forming silicon dies with structural dimensions of  $10\text{ }\mu\text{m}$  and less have been used (fig.1). The micro-structures could be moulded in different materials using both cold and warm (superplastic) forming (fig.2). The precision and surface quality of the formed parts correspond to the high quality of the micro-structured die. Both the low surface roughness and the accurate edges of the silicon structures are represented in the moulded structures. In cold forming attempts the dies had to withstand very high compressive stresses ( $2000\text{N/mm}^2$ ). Depending on the work piece material die wear or even die failure could be observed. Whilst damages were small during embossing of aluminium often die failure occurred during forming of stainless steel parts. Very good results could be achieved in superplastic forming of Zn78Al22. Due to the low compressive stresses at forming temperatures of  $250^\circ\text{C}$  no die wear occur.

The results show that the use of etched silicon dies for microforming enables the accurate moulding of smallest structures in different materials. However, due to the high brittleness and tensile stress sensitivity the range of use of silicon is apparently limited to soft materials and/or low compressive stresses.

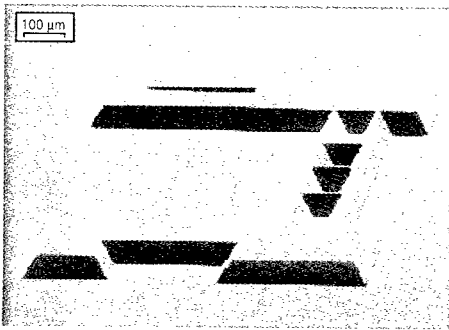


Figure 1: silicon die with micro-structure

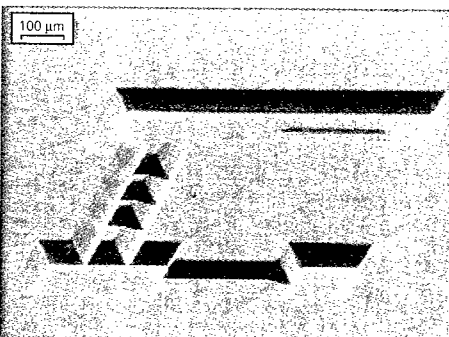


Figure 2: embossed structure in Zn78Al22

**Outlook**

To define the limits of silicon for microforming dies more exactly further investigations on the influence of coatings as well as die and micro-structure design will be necessary. Furthermore other die materials and structuring technologies have to be found meeting the high requirements concerning precision, surface roughness, strength, and ductility.



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## Experimental determination of the correlation of conductivity and mechanical stress of a silver-filled electrically conducting adhesive

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In practice electrically conducting bonded joints between surface-mounted devices (SMD) and substrates are qualified by experimentally examining the complete setup at low and high temperatures as well as under the influence of moisture. Based on these results, however, it is hardly possible to design micro bonds with modified adhesive layer geometries, differing substrates or adhesives. Therefore a simple and cost-effective testing procedure which allows a reliable analysis of arbitrary electrically conducting bonded joints should be developed.

Due to the combination of mechanical stress and thermal strain a multi-axial state of stress exists within the electrically conducting adhesive layer which can initiate functional and structural failure of the bonded joint. Analysis using the Finite Element Method seems to be the only feasible way to determine the multi-axial stress state within the adhesive. A prerequisite for the mathematical modelling, however, is reliable material data for stiffness and strength. Also, the correlation between mechanical stress and electrical conductivity of a joint has to be taken into account in order to be able to predict functional failure.

For a combined examination of mechanical and conducting material behaviour it is necessary to extend the usual mechanical testing methods for bonded joints. One possibility is the modified thick-adherend tensile test following DIN 54451 and ISO 11003-2. The specimens are made from steel adherends which are connected, electrically conducting, with silver-filled adhesive. In a four-wire resistance measurement using an high precision ohmmeter very low basic electrical resistance between the steel adherends of the lap-shear joint can be measured. In a shear strain-rate-controlled test with electrically mounted specimens it is possible to determine the load-strain behaviour against small changes of resistance, thus describing a relationship between mechanical stress und electrical conductivity. In biaxial tests where butt-bonded hollow cylinders are used the change of resistance can be correlated even with multiaxial stress states.

## Delay-time sensors for electrodiffusion measurements

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The electrochemical flow measurements are based on the detection of the amplitude from electrodiffusion current of a working microelectrode. This type of probes or «Amplitude Electrodiffusion Sensors» involves current amplitude as the result the main information parameter of AED sensors like the wall velocity gradient  $S = (\partial V / \partial y)_{y=0}$  or the wall shear stress  $\tau = \mu S$ , where  $\mu$  is the dynamic viscosity. Among the main limitations there are two disadvantages. Firstly the results of flow measurements by means of these probes provide either relative evaluations or there are rather instable and necessitate to calibrate all sensors before the experiments. The second inconvenient is one to be constrained to use special electrolytes, for instance the solution of  $\text{Fe}(\text{CN})_6^{3-} / \text{Fe}(\text{CN})_6^{4-}$  and  $\text{I}_3^- / \text{I}^-$  red-ox systems. These difficulties restrict the application of the AED sensors.

Another possibility to extend the field of applications of electrochemical flow measurements is to employ the microelectrode as «Delay-Time Sensors» or DTS technique. This new version allows us to overcome the previous restrictions and is capable to supply an alternative electrochemical diagnostic of flows in laboratory testing conditions, in some industrial processes and even natural conditions.

Indeed, the technique can be applied in some industrial and natural electrolytes, as in sea water conditions. In that context the special design of ED sensors one may dispose two closely spaced working electrodes: anode (or generator electrode) and one microelectrode (cathode or detector electrode); the red-ox system employing natural choice-ions  $\text{Cl}^-$ . Consequently the electrochemical reaction of chlorine reduction and electrical current  $I_D$  arises in the electrical circuit of the detector electrode. This current arises some delay time  $T$  after the step polarisation of the detector electrode due to the diffusion of the chlorine molecules from the generator electrode  $I_G$  to the detector one  $I_D$ . The diffusion process is controlled by the flow conditions and together with the delay-time  $T$  measurements give us information about near wall hydrodynamics.

Therefore the aim of the present paper is to provide a delay-time  $T$  prediction for the non- stationary diffusion equation as following

$$\frac{\partial C}{\partial t} + Sy \frac{\partial C}{\partial x} = D \frac{\partial^2 C}{\partial y^2} \quad (1)$$

where  $C = (x, y, t)$  and  $D$  denote respectively bidimensional concentration and diffusivity of the molecular chlorine. We suppose that the characteristic length of the probe  $L$  is small so as the wall shear stress value  $\tau$  in the equation can be processed as the local one.

The boundary conditions at the electrodes set in-the solid surface  $y = 0$  for time  $t > 0$ :

$$0 < x < L; \quad \frac{\partial C}{\partial y} = \frac{I_G}{LD}; \quad L < x < 2L; \quad \frac{\partial C}{\partial y} = 0; \quad 2L < x < 3L; \quad C = 0 \quad (2)$$

The resolution of equation (1) depending on the associated conditions (2) lead to

$$T = b \left( \frac{L\mu}{\sqrt{D}} \right)^{\frac{2}{3}} \tau^{-\frac{2}{3}} \quad (3)$$

where b is a constant. Our result is in good agreement with the experiments of the delay-time electrochemical sensor T used by some authors.

## Anomalous trends in electronic properties of boron compounds BP, BAs, and BSb

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We have performed an ab-initio investigation for a series of boron compounds, BP, BAs, and BSb, and have compared their structural and electronic properties with those of c-BN. The calculations are performed using a planewave expansion within local density approximation and the pseudopotential approximation. Results are given for lattice constants, bulk moduli, band structures and band-gap pressure coefficients. The electronic band structure of these compounds showed to have features that differ from those of other III-V materials. We found that the direct band-gap pressure coefficient in boron compounds is nearly independent on the anion substitutions. As a result, this trend is similar to the one caused by the cation substitutions in other zinc-blende compounds. This is another anomalous case which can be characterized by reversing the standard assignments for the anion and cation in these compounds. The essential features of pressure-induced changes in electronic band structure are exhibited from the ab-initio results by two characterization parameters: the potential deformation, and the band-gap pressure coefficients.

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## Dynamic mechanical analysis of an high-filled epoxy resin in thin layers

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Epoxy resins are important materials for microelectronics. They are used for underfilling, as glop top encapsulation and as die adhesives. For exact simulation of the reliability of microelectronic components, it is necessary to know the real mechanical properties of each material. As the properties of macroscopic samples are not identical with characteristics of microscopic test specimens, the mechanical properties of materials used in microelectronics have to be determined in the actual application thickness.

This report presents the results of an dynamic mechanical analysis (DMA) of an high-filled epoxy resin in three different layers (1000  $\mu\text{m}$ , 500  $\mu\text{m}$ , 100  $\mu\text{m}$ ) to demonstrate the dependence of the typical mechanical characteristics of polymers on the sample thickness. For these experiments a 3-point bending system was used. One of the emphasis of this work was the sample production and preparation. The samples were casted between two parallel glass plates with a defined gap and cured. Then the specimens were isolated in bars by laser cutting or sawing. Very important points with regard to precise measure values in these process were the exact sample geometry, a good distribution of the filler and the quality of the cutting surfaces relative to the geometry and micro cracks. Material properties like Complex Modulus, Storage Modulus and Loss Modulus were determined with DMA in dependence on the temperature and the glass transition temperature. The results of these measurements show that with decreasing layer thicknesses the Modulus increases and the mechanical characteristic should always be measured as close as possible to the real application thickness.

## Some remarks about indentation tests with spherical indenters

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In the present paper some effects influencing the force-displacement-curves and displacement fields of indentation tests are discussed. The force-displacement-curves are of interest in order to determine materials parameters.

Spherical indenters are non-selfsimilary and so they have the advantage, that during indentation tests different characteristic strains in the specimen occur. Therefore the information of the whole stress-strain-curve of a material is contained in the force-displacement-curve. But there are some other influences too like friction between indenter and sample surface, kinematic hardening or creep. In order to avoid errors in the determination of the material parameters it is necessary to take additional available information into account.

An important point is the surface profile after unloading including the effects of piling up and sinking in outside the indentation area. Information about friction can be obtained by cycling load/unload calculations or experiments.

In this work some numerical results are compared with corresponding experiments on steel samples with known stress-strain-curves. The piling-up was measured with an Atomic Force Microscope (AFM) and the ratio of real and calculated contact area was obtained. This value is also important for the accuracy of hardness results.

The results show, that experimental findings can be confirmed by the numerical results using a careful selection of the input material parameters. This allows for the future a better analysis of experimental data.

## Silicon three-axial tactile sensor for micromaterial characterization

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- micromaterial characterization/advanced mechanical testing methodes

A three-axial tactile force sensor for the investigation of micromechanical structures has been developed using silicon micromachining technology. The sensor is capable of performing micro material characterization such as the determination of the spring constant of micromechanical structures. Another application for this sensor is dimensional metrology where it has been tested as a 3-D probe in a micro coordinate measurement machine.

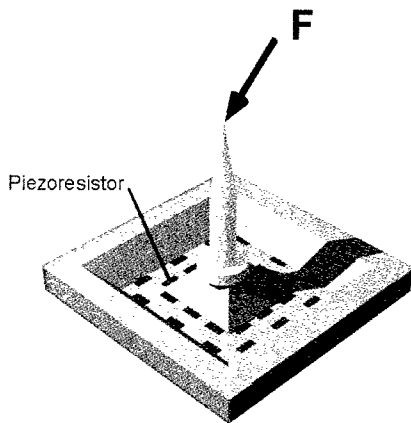


Figure 1: Schematic view of the 3d tactile force sensor

The sensing element of the sensor consists of a boss-membrane which has been fabricated through bulk silicon micromachining by a process compatible to standard CMOS technology (Fig.1). The square opening is  $3 \times 3 \text{ mm}^2$  and the membrane is  $20 \mu\text{m}$  thick. The tactile element of the sensor consists of a stylus which is fixed to the boss by epoxy resin. The stylus is  $500 \mu\text{m}$  in diameter and  $4 \text{ mm}$  long. Standard processing steps for insulation, strain gauge and metallization are then carried out to build 3 Wheatston bridges on the diaphragm each detecting one of the three components of the applied force vector. FEM-simulations have been performed to minimize the cross talk between the three channels

of the sensor by varying the arrangement of the resistors on the diaphragm. A calibration of the sensor has been performed using a new micro force detection system which consists of a 3-D coarse positioner, a fine 1-D positioning system and a compensation balance. Sensitivities of  $0,014 \text{ mV/V/mN}$  for the z-direction and  $0,05 \text{ mV/V/mN}$  for the x and y-direction have been measured. Regression of the sensor output against the applied force shows excellent linearity ( $r^2 = 0,998$ ). The described sensor has been successfully used to measure the spring constant of a cantilever fabricated by standard bulk silicon micromachining (Fig. 2/3).

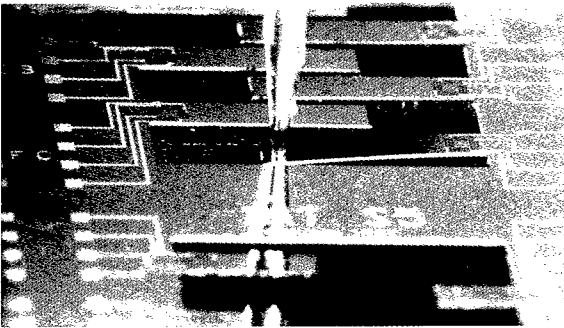


Figure 2: Deflection of a silicon cantilever using the tactile force sensor

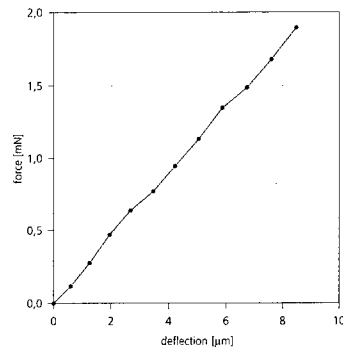


Figure 3: Spring constant measurement of a silicon cantilever

Used as 3-D probe in a micro coordinate measurement machine the sensor shows the following characteristics:

	x-y-direction	z-direction
<b>resolution</b>	3 nm	5 nm
<b>repeatability of contact point position</b>	10 nm	20 nm
<b>stiffness</b>	2.5 mN/μm	55 mN/μm
<b>crosstalk x-y-direction</b>	15 nm/μm <sub>x</sub>	-
<b>crosstalk z-direction</b>	15 nm/μm <sub>x</sub>	-

For a maximum deflection of 3 μm and a fast deflection velocity of 100 nm/s we found in all cases a high linearity of the force-displacement curves and no hysteresis deformation. With deflections up to 60 μm in x-y-direction and a very slow deflection velocity of 100 nm/min we detected hysteresis deformation due to the relaxation of the epoxy resin. The maximum deflection in z-direction should not exceed 10 μm because of possible mechanical destruction of the boss membrane.

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## Micromechanical actuators based on shape memory effect

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The ongoing trend towards reduction of size, weight, and power consumption in micro- and nanotechnology, microsurgery, and bioengineering requires the availability of intelligent and adaptable systems of micromechanical actuators. Therefore, worldwide research has been started in order to develop new actuator concepts and materials.

Shape Memory Alloys (SMA) have the potential to become a material of great importance in microactuator technology. The shape memory effect, exhibited by several materials such as NiTi alloys, is based on the thermally induced crystalline transformation between a low temperature martensite phase and a high temperature austenite phase. It offers significant advantages compared to other actuator concepts: high energy density, various kinds of movement, high design flexibility, and easy actuator control by direct electrical current heating. In microactuators, the response of SMA can be enhanced appreciably because the extreme surface-to-volume ratio of microstructures leads to a strongly increased heat transfer to the surrounding. In addition, the dynamical behaviour of SMA actuators can be further improved by using a differential-type actuator configuration.

This paper reports on two SMA microactuators which are the technology drivers for our efforts at Braunschweig, a micromachined gripper with flexure hinges and a multi-actuator system based on the artificial muscle principle. In both devices, NiTi foils with a thickness of 50  $\mu\text{m}$  are used as actuators. The foils are micromachined using a Q-switched Nd:YAG laser at a wavelength of 1064 nm. In a subsequent wet chemical etching process, the heat affected zone is removed in order to improve the mechanical properties of the elements [1]. Furthermore, the transition temperature between the austenite phase and the martensite phase depends on mechanical stress. Therefore, in order to obtain a distribution of stresses over the SMA elements as homogeneous as possible, it is necessary to optimize the shape of the microactuators. This can be achieved using the Finite Element Method.

The gripper developed for microassembly purposes is an improved version of a microgripper described elsewhere [2]. It is fabricated by photolithography and fast anisotropic silicon etching and is driven by a differential-type SMA actuator. Mounting of the actuator elements is achieved by clips plugged into the silicon element which leads to a positive connection.

The multi-actuator system is composed of numerous identical microactuators. In order to fulfil great demands on SMA actuators regarding force, displacement and flexibility in movement, the single microactuators are connected in parallel as well as in series, corresponding to the basic structure of a natural muscle. Based on the results of a feasibility study using enlarged models [3] the artificial muscle system consists of identical silicon elements each driven by a differential-type SMA actuator.

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## Interfacial engineering in nanocomposites

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This paper will present details of interfacial design, structure and properties in a variety of different nanocomposites. The materials discussed include co-sputtered nanoscale Ag/SiO<sub>2</sub> and Ag/Si thin films which exhibit novel optoelectronic behaviour, and melt spun nanoscale Pb/Al and Sn/Al sheets which exhibit novel mechanical behaviour. The relationships between processing, structure and properties are discussed for these unusual materials systems.

## The material properties, growth technology and applications of wide bandgap semiconductors for sensors and electronics

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The last decade has seen enormous advances in the growth of wide bandgap semiconductor materials and their application in a growing range of electronic devices. This talk will review some of the developments in silicon carbide, the III - nitrides and diamond and will endeavour to highlight the existing challenges and opportunities for exploiting these wide bandgap semiconductor materials.

Silicon carbide has been the focus of considerable attention for high power, high frequency and high temperature electronics. Although the device processing of silicon carbide shares many common features with silicon technology, for instance it has a complementary oxide, the development of silicon carbide substrates is still plagued by significant micropipe defect densities [1]. Nevertheless advances in chemical vapour deposition, compensation doping of intrinsic carrier concentrations, dry etching techniques and advanced metallisation schemes have made it feasible to manufacture high performance power devices and sophisticated microsystem components fabricated in silicon carbide.

The status of the III-nitride materials, such as gallium nitride and its related compounds, is still in its infancy compared with silicon carbide however the recent strides made in blue light emitting diodes and lasers will inevitably feed progress in a widening range of other nitride based device technologies. For example, the progress in lateral overgrowth processes has enabled the deposition of III-nitrides epilayers with dislocation densities low enough to achieve estimated laser diode lifetimes of 10,000 hours [2].

The suitability of diamond as a semiconductor material has been contentious. The identification of sulphur as an n-type dopant in diamond [3] with an activation energy closely matched to the boron acceptor in diamond is set to decide the issue of diamond for active electronic applications. Regardless of this scientific debate, diamond has succeeded in finding commercial applications as a day-light blind UV detector material and also in high frequency surface acoustic wave devices. The future potential for diamond electronics will be reviewed within this rapidly changing context.

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## BGA, flip chip and CSP solder joint reliability

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Accelerated Thermal Cycling (ATC) results show that solder joints of most CSP assemblies fail earlier than those of conventional leaded or BGA assemblies. For example, many CSP assemblies fail in a few hundred cycles of thermal cycling between 0 and 100 °C. Whether this is acceptable or not depends on the use conditions and the intended product design life. Since safety margins appear to be reduced for CSP solder joints, interpretation of failure distributions under test conditions and extrapolation of test results to field conditions require accurate life prediction models and acceleration factors.

This paper addresses these issues by comparing conventional SMT, BGA and CSP test results, and deriving acceleration factors for a variety of packages and assemblies. Several life prediction models are used to estimate acceleration factors. Failure data are then extrapolated to common field conditions to facilitate the interpretation of test results in terms of the useful life of circuit board assemblies. Validation of life prediction techniques is discussed as well as reliability challenges posed by the coming of age of lead-free assemblies.

## Microstructural characterization of Al/SiC interface after thermal treatments by TEM, X-ray diffraction and AFM

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The aluminium composite materials reinforced with SiC particles has shown good physical properties, manufacturing flexibility and low cost in the field of electronic packaging. There is still a need to better understand the microstructural factors which are associated with the thermal conductivity of these advanced materials, with special focus on the role of the particle-matrix interface.

Lattice distortions measured by X-ray diffraction line broadening Fourier analysis, TEM and AFM observations are correlated after different thermal treatments with the thermal conductivity in order to better characterize the SiC/Al interface bond.

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## Nano-writing with atomic force microscopy

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Atomic force microscopy and electrical conductivity microscopy have been used in controlled environments to modify thermally grown oxide layers of known thickness in the range 2.4-6.4 nm. A tip-to-substrate bias of greater than 5 V, with the substrate positive, leads to oxide growth at the contact point. The growth rate is exponentially dependent on the applied bias for any given initial oxide thickness. The growth rate is also weakly dependent on dwell time (scan rate). At positive bias less than 5V the original oxide is removed.

The growth mechanism requires: i) a source of oxidants; ii) transport of the ionic oxidants to the Si/oxide interface, and; iii) oxidation at the Si/oxide interface. The proposed growth mechanism will be discussed in detail together with the proposed mechanism for material removal.

These results and proposed mechanisms present considerable opportunities in the areas of nano-lithography and ultra-high density digital data storage.

## Thermotropic metal-containing liquid crystal polyurethanes, possessing the high electric conductivity

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Metal addition to a polymer chain is a promising way of polymer modification. One of the well-known techniques for preparation of metal coordinated polymers is the polymerization of monomer containing metal complex. When monomer takes part in the coordination with metal salt, that effects its chemical structure and then influences the polymer matrix formation and the properties of the polymer.

Metal-coordinated polyurethane oligomers having thermotropic liquid crystalline properties were obtained on the basis of polynuclear complexes of 3d-metals, symmetrical aromatic diisocyanates and polyoxyetheleneglycols.

Two ways were used for synthesis of liquid crystalline oligomers. In the first case rigid aromatic blocks are connected as results of the formation of complexes by urethane groups. In this process a rather wide range of the existence of mesophase is observed. The specific resistance of these samples at temperature range 20 – 90 °C is  $10^8 - 10^6$  Ohm·cm.

The mechanism of the electron transfer in this case is connected with a jump of the carrier from one 3d-metal coordinated fragment to the other one by electron-donor groups being part of the flexible spacer.

In the second case mesogenic structure is a »stack« consisting of azoaromatic blocks connected by the coordination by metal ions.

In this process the temperature range of the existence of mesophase narrows markedly, but the electron transfer proceeds through the »stack« in much the same way metallophthalocyanine.

The specific resistance of this mechanism of the transfer at the temperature reange 20 – 90 °C is  $10^5 - 10^2$  Ohm·cm.



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## Damage modelling via bi-modal surface energies

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A new model for damage has been proposed recently by L. Truskinovsky and the present author [1], [2]. It is based on the separation of the elastic energy into two parts, bulk and surface, just as in the approach to fracture mechanics introduced by Griffith (1920) and modified later by Barenblatt (1959). Within this common approach, the difference between fracture and damage lies in the shape of the surface energy, which in both cases is assumed to be a function of the amplitude of the crack opening. In the case of fracture, this function is concave, while in the case of damage it is bi-modal. This second type of function is a monotonically increasing function exhibiting two plateaus, one for small and the other for large values of the crack opening. The two plateaus are responsible of diffused micro-cracking and of terminal macroscopic failure, respectively.

In the presence of a bi-modal surface energy, the minimization of the total elastic energy in a condition of increasing elongation predicts a progressive formation of cracks, one after the other. This type of response was observed in experiments performed by Elam (1938) and Mc Reynolds (1949), and, more recently, by Royer and Froli (1997). Damage is clearly identified in the resulting stress-strain curve, and its intensity is measured by the number of cracks.

The proposed model leaves some indeterminacy in the conditions for the creation of a new crack; the indeterminacy is related to the capability of the system of overcoming energy barriers when in a metastable equilibrium configuration. Unloading curves at various stages of the damage process may also be determined, at the price of switching from global minimization to stepwise minimization, i.e., from a global equilibrium problem to a sequence of incremental equilibrium problems.

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## A review of fully-depleted SOI CMOS technology for microsystems

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### INTRODUCTION:

Based on an extensive review of the material, process, device and circuit properties of thin-film fully-depleted (FD) SOI CMOS, our work demonstrates that this technology emerges as the most promising and mature contender for integrated microsystems which must operate in the temperature range 200-350 °C.

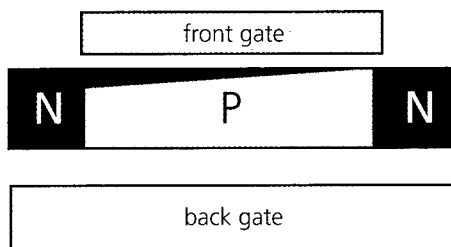


Figure 1:  
Cross-section of a thin-film FD SOI n-MOSFET, showing depletion region extension (grey) under channel (dark) and drain junction area (large dashed line), in the active Si film surrounded by its front and back oxides and gates.

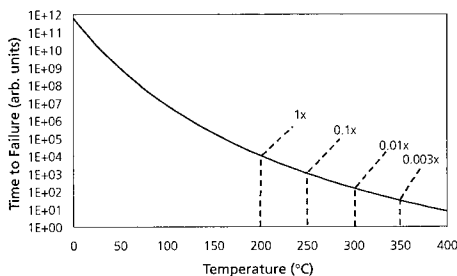


Figure 2:  
Time to failure with arbitrary units

due to electromigration. Tungsten (W) is presently considered as the best metal candidate. However, Tungsten reacts with silicon to form silicide during annealing processes. Thin SOI devices may be destroyed if all the silicon is consumed in the contact area, leading to very large series resistance. To prevent this, a simple self-aligned barrier is

### TECHNOLOGY:

Our research is focused on a 2µm FD-SOI CMOS process on either SIMOX or more recent SmartCut UNIBOND wafers which is compatible with standard Bulk CMOS. The thicknesses of the layers are 400 nm for the buried oxide, 80 nm for the silicon and 31 nm for the gate oxide (Fig. 1). This process presents optimum electrical properties for operation up to 300 °C due to the reduced film thickness and threshold voltage (0.7 V at 25 °C, still above 0.3 V at 300 °C). The transistors work up to 350 °C but with degraded performance.

### RELIABILITY:

An important advantage of our technology which enables very high temperature operation is to significantly accelerate circuit failure analysis. For example, assuming the Arrhenius law is valid, analysis time at 350 °C is 300 times smaller than at 200 °C (Fig. 2). In order to allow for accelerated circuit lifetime analysis and reliability characterization at such high temperature, on-chip Al-metallization has to be substituted

formed on the source, drain and gate regions. We have demonstrated the efficiency and reproducibility of  $\text{TiSi}_2/\text{CoSi}_2$  sandwich contact barriers on very thin SOI films.

### DEVICES:

Si MOSFET characteristics are significantly affected at high temperatures, by the drift of threshold voltage  $V_{th}$  and the increase of junction leakage current  $I_{js}$  [1]. In bulk Si and partially-depleted (PD) SOI MOSFETs,  $V_{th}$  decreases with depletion width by 2 to 5 mV/°C and  $I_{js}$  is proportional to drain junction area ( $A_{jd}$ ) and  $n_i^2$  since dominated by diffusion mechanisms of excess carriers in the quasi-neutral transition regions. We have observed that typical bulk  $I_{js}$  rises to about 0.1  $\mu\text{A}$  per  $\mu\text{m}$  of device width at 250°C. In PD SOI, thanks to dielectric isolation,  $A_{jd}$  is typically two orders of magnitude smaller than in bulk and so is  $I_{js}$ . In FD SOI, the depletion extension being equal to the film thickness, it remains constant with temperature and  $V_{th}$  shifts are reduced to 0.7 to 1.5 mV/°C. Furthermore, as quasi-neutral regions are suppressed, only generation mechanisms may contribute to  $I_{js}$  which is then proportional to  $n_i$  and the depletion region volume and hence, almost three orders of magnitude lower than in bulk. Finally, we have also demonstrated that optimized SOI lateral bipolar transistors, compatible with thin-film SOI CMOS process, may present sufficiently low leakage currents and good performance as required in high temperature applications incorporating voltage and current references.

**DIGITAL CIRCUITS:** On the circuit level, device leakage current will directly contribute to standby power dissipation. Extrapolating individual device leakage towards more than 1-million transistor on-a-chip future high-temperature applications, we stress that with bulk Si technology, more than 1 W of power would have to be dissipated above 250 °C, which does not seem acceptable. This was confirmed by experimental 64 kb SRAM results [2]. Thanks to its lowest leakage, FD SOI CMOS therefore enables the realization of complex electronic circuits to be operated above 250 °C.

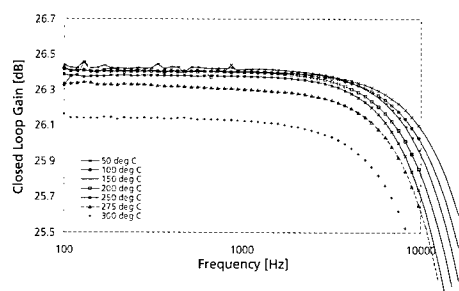


Figure 3:  
Measured frequency response of our instrumentation amplifier from 25°C to 300°C.

**ANALOG CIRCUITS:** Our lab has presented efficient high temperature analog circuits since years. It includes an instrumentation amplifier with integrated N-doped diffusion resistors which shows a constant gain of 21 from 25 °C to 250 °C with only 0.45% deviation (Fig. 3). MOSFET-C whose frequency response can be held constant from 25 °C to 300 °C with an automatic tuning have shown good distortion results ( $<-50\text{db}$  on 3V supply and  $V_{in,pp}=0.5\text{V}$ ). Bandgap voltage references using SOI lateral bipolars with temperature coefficients better than 100 ppm/°C up to 300 °C have also been

realized. Our latest result concerns a second order SIGMA-DELTA analog to digital converter with switched capacitor integrator showing a maximum SNR of 60dB and a dynamic range of 11 bits at room temperature. Resolution at 250 °C is still 9 bits. The circuit shows functionality up to 350 °C.

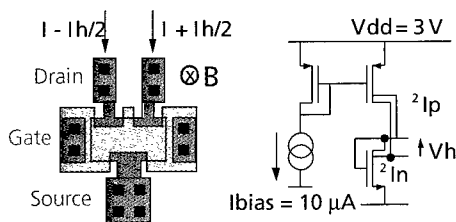


Figure 4:  
Magnetic field split-drain sensing MOSFET and circuit.

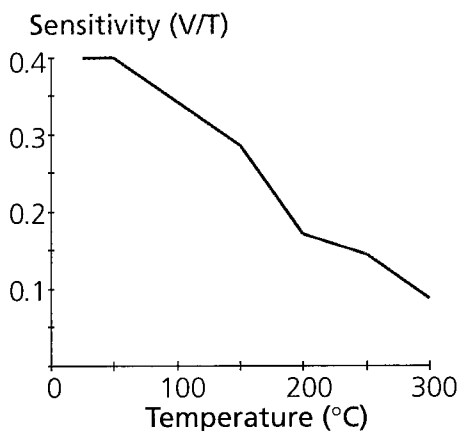


Figure 5:  
Measured Hall sensor sensitivity as a function of temperature, for a 0.01 mA – 3 V power supply.

**SENSORS:** High-temperature magnetic sensors are needed in several applications such as position sensor in aeronautics, motor magnetic features and regulation... Bulk MOS split-drain Hall sensors are well known for good performance and CMOS process compatibility. SOI devices offer additional advantages such as better impurity profile uniformity and extended temperature range. The split-drain MOSFET sensor is shown in figure 4. In the associated circuit, the drain current imbalance of the two complementary split-drain n- and p-MOSFETs are added at the high impedance output node and converted to voltage. The sensitivity has been measured as a function of temperature (Fig. 5), the decrease being mainly related to mobility reduction. Taking power dissipation and temperature range into account, this corresponds nevertheless to fairly good sensitivity results when compared to conventional Si or GaAs sensors, typically yielding about 1-10 V/T but for 1-10 mA and 5-10 V power supplies and temperature range limited to 150 °C.

The SOI lateral bipolar bandgap references also constitute the basic building block for achieving temperature measurement sensors correctly operating up to 300-350°C.

## CONCLUSION:

Our work demonstrates that thin-film fully-depleted SOI CMOS technology emerges as the most promising and mature contender for the temperature range 200-350 °C, since it permits, even at elevated temperature, low-voltage low-power performance, VLSI integration, modeling/design/CAD compatibility, noise immunity, process simplicity and scalability... To support this analysis, we have realized FD SOI CMOS circuits which operate up to 300 °C or more and implement one-chip ASIC analog instrumen-

tation chains involving magnetic and temperature sensors, amplification, filtering, A/D conversion, logic gates... The availability of tungsten metallization will assure the industrial feasibility and usefulness of the FD-SOI technology for High Temperature applications thanks to enhanced circuit reliability and faster failure analysis procedures.

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## Texture and internal stresses in FeCo – layers for microinductors

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Texture and internal stresses are known to influence the physical and the mechanical properties of components significantly. In case of small microelectronic devices especially the texture and the residual stresses in thin multi-layers are of interest with respect to the adhesion of the layer to the substrate and the magnetic properties. A non-destructive analysis of the texture and the residual stresses in these thin layers is possible using diffraction methods.

Here, the texture and the residual stresses in sputter deposited  $\text{Fe}_{60}\text{Co}_{40}$  mono layer and multi-layers as well as in  $\text{Fe}_{50}\text{Co}_{50}$  mono and FeCo-  $\text{SiO}_2$  - multi-layers was determined using X-ray diffraction. The results of the experiments reveal a strong dependence of the texture as well as the residual stresses on the chemical composition and the thickness of the mono layers. In case of the multi-layers additional parameters are the thickness of the individual mono-layers as well as the number of mono-layers. Further on the texture and the residual stresses depend on the parameter of the deposition process such as the sputter conditions such as pressure and the power.

The texture and the residual stresses determined will be discussed in relation to the performance of the microinductors.

## Water in subnanometer-size local free volumes of polyimides and polyamides: A positron lifetime study

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Polyimides have found widespread application as gas separation membranes and as insulating layers in microelectronic device fabrication. A major drawback of these polymers is the moisture absorption which leads to swelling of the material, thus degrading its electrical properties. Although of great importance, the exact mechanisms that govern humidity-induced water uptake are still unclear. It is generally believed that both sorption and swelling behaviour are closely related to the free-volume properties. The study of local free volumes which occur due to the structural disorder in amorphous polymers is a new field of application of positron method. Due to a lack in experimental techniques for investigating subnanometer-size holes, this method is of increasing interest.

We use positron annihilation lifetime (PAL) spectroscopy to study the effect of water uptake on the free volume in 6FDA-ODA polyimide. Ortho-positronium (o-Ps) lifetime ( $\tau_3$ ) and intensity ( $I_3$ ) data were analysed assuming a Gaussian size distribution of pre-existing (excess free-volume) holes (estimated mean hole volume  $v = 0.134 \text{ nm}^3$ ), and further assuming that holes occupied by water molecules would not be detected in the measurement. When exposed to humidity, both the mean hole volume and the number of holes not occupied by water molecules decreases with increasing relative humidity, as indicated by decreasing  $\tau_3$  and  $I_3$ . Our PAL results correlated with humidity-induced mass uptake and volume expansion support a model according to which water absorption in polyimides occurs in two stages. At relative humidities smaller than about 30 %, water is absorbed mostly in large, pre-existing holes, with each hole typically occupied by a single water molecule. At larger humidities, an increasing fraction of the sorbed water molecules will occupy sites other than pre-existing empty holes. A multiple occupation of larger holes by water molecules is discussed as a possible mechanism.

The permeability/selectivity ratio of polyimides can be improved by physically modifying the membrane structure using ion irradiation. We studied Kapton polyimide prepared by spin-coating using a silicon wafer as the substrate. The films were bombarded with  $1 \times 10^{15} \text{ B}^+$  ions/cm<sup>2</sup> (180 keV). This treatment resulted in an increase in the maximum mass uptake from 3.9 % to 14 % at 100 % RH of the ambient atmosphere. The PAL investigations provide evidence that  $\text{B}^+$  bombarding of Kapton leads to the formation of a large density of small ( $v = 0.040 \text{ nm}^3$ ) holes in the material.

Others than polyimides, polyamides (Nylon 6.6) showed an increase in the mean hole volume due to ~8 % water uptake. This effect can be explained by plastization of Nylon and decrease of the glass transition temperature due to humidity sorption.

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## Submicroscopic and atomic scale structure changes during decomposition of Al alloys: A positron annihilation study

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Al alloys represent an important engineering material for transportation (aerospace, automotive) technologies. Special Al alloys are also used for manufacturing microwires applied for bonding microelectronic devices. Most of the supersaturated Al alloys decompose during post-quench aging by forming a sequence of metastable phases. These metastable phases when precipitated into a fine structure of nanometre-size coherent or incoherent particles improve distinctly the mechanical properties of this class of materials. It is an important aim of the research to understand the sequences and the nature of the metastable phases occurring. This is particularly true for the early stage of precipitation. For this purpose experimental techniques which are sensitive on an atomic scale are of great interest. Usually, these techniques (such as high resolution electron-microscopy HRTEM, and atom probe-field ion microscopy AP-FIM, for example) are very expensive and can not be applied for non-destructive testing.

Positron annihilation techniques are relatively cheap and can potentially be applied for non-destructive testing [1]. We will show that the combination of positron annihilation lifetime and Doppler-broadened annihilation line measurements provides a unique tool for studying structural (microscopic and atomic) defects and variations in the chemical surroundings of these defects. Positrons response to semicoherent and incoherent precipitates, but also to alloy atom cluster as small as 20 atoms and to coherent GP zones [1].

Laboratory alloys of Al-Cu and Al-Cu-Mg and the corresponding commercial alloys 2021 and 2024 were studied [1,2]. After solution annealing and quenching of Al-Cu alloy excess vacancies bound to Cu atoms or Cu atom clusters were observed. During artificial ageing imperfect Guinier-Preston zones may be formed which contain also vacancies. The presence of both Cu and Mg atoms in the neighbourhood of quenched-in vacancies was clearly detected in the Al-Cu-Mg alloy. In a later ageing state, semicoherent  $\theta'(S')$  and incoherent  $\theta(S)$  particles trap positrons. The presence of Mn in the commercial alloys modifies the precipitation structure in a characteristic manner [2].

As-quenched Al-Si1at.% alloy used for manufacturing microwires showed small vacancy agglomerates (2 - 4 missing atoms) bound to Si atom clusters [3]. During annealing up to 180 °C the agglomerates form small Si precipitates. At 250 °C a change in

the microstructure of the Si particle-matrix interface is observed. Dislocations produced by plastic deformation of AlSi1 form nucleus for Si precipitation. This results in a very finely dispersed Si precipitates structure which leads to a distinct improvement of the mechanical properties of the alloy.

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## Internal stresses and life time evaluation of MEMS

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In this study, we optimized the strain induced by PECVD (Plasma Enhanced Chemical Vapor Deposition) low stress isolating layers on silicon wafers. The influence of the strain and stress on aging is reported. Life time evaluation is correlated to the aging of MEMS. PECVD (Plasma Enhanced Chemical Vapor Deposition) is a well known process to fabricate thin isolating layers for electronic structures. The concept of the "Plasma-Box" of the Nextral D 200 PEVCD system permits to fabricate low stress isolating layers especially for micromechanical parts and microsystems. Unlike the conventional PECVD processes, which are based on compensating multilayers of tensile and compressive strained layers, the »Plasma-Box« system allows to produce single layer structures with lower stress values. As produced layers are of major interest to sensor manufacturers, when it comes to accurately control stress and to obtain very high breakdown voltages on silicon or metal substrates. Efficient in-situ plasma cleaning of this batch tool is another advantage that places the system in a very favorable market position.

A high-resolution X-ray diffractometer (HRXRD) allows measuring the strain of a crystal. This is an accurate; non-destructive microscopic method applied in the field of MEMS to obtain quantified results on the strain. Aging of a micromachined silicon actuator results in a change of the strain profile. The method is applied to actuators on which hysteresis of displacement was observed. It is shown that the sensitivity of HRXRD is accurate enough to detect even small stress on cantilevers before a bending could be observed. In addition, the technique of reciprocal space mapping (RSM) developed in recent years has become a valuable tool for obtaining more detailed information on thin layers. We show that the more detailed information provided by the RSM leads to even more accurate results than the HRXRD measurements on the thin isolating layers.

## Lead-free solders - rapidly reflowed under the laser beam

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As decreed by the EC regulation »Waste from Electrical and Electronic Equipment«, beginning with 2004 in Europe the ban on lead in electrical, electronical, and technical products will become valid. The toxicity of lead and its alloys is one of the reasons for the close substitution. But in addition the demands concerning to the mechanical, thermal, and electrical characteristics of solders and solder joints are increasing. Besides to the common requirements completely new ones are claimed. This is due to new operation fields along with processing temperatures up to 180 °C and even more. Higher processing temperatures and power cycle strains generate interfacial damages. They also induce a decrease of cohesive forces because of inappropriate fatigue behaviour and insufficient thermal properties of conventional, leaded solders. So, today the most considerable requirements on solder and soldering joints are higher strength as well as long-term- and fatigue stability. The latter ones mostly depend on the joints' structure, i.e. the possibilities of receiving or keeping it in a microcrystalline configuration. Grain refining by rapid reflowing is an innovative technology for improving the structure based characteristics of solders. The procedure of rapidly reflowing special lead-less soldering metallizations or rapidly solidified solder foils changes their attributes in an incredible way. After optimising all technological parameters of laser-beam reflowing, the hardness of the reflowed areas increases notable. As a result rapid reflowing under the influence of continuous laser pulses enables significant higher strength of joints which are laser-beam soldered afterwards. On basis of various experiments uniform low-faulty quality of soldered joints with higher strength as well as improved long-term stability has been verified. With the quality extension of lead-less soldering system and joints through crystal refining by rapidly reflowing a new promising technological approach is available to meet urgent demands of the electronics industry.

## UNIDAC - cross correlation based deformation analysis at digitised micrographs to study material behaviour and parameters in MST

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Correlation analysis of grey scale patterns is a method that allows deformation field measurement in digitised images of the objects under investigation based on algorithms of digital image processing. Measurement of displacement is based on - at least - two images which are compared one to each other. This comparison is performed by means of two-dimensional cross correlation analysis applied to a set of local intensity submatrices taken from the pattern matrices of the reference and the comparison image in the surrounding of predefined measuring points. Subpixel accuracy down to 0,25 .... 0,01 pixels is obtained using special so-called subpixel algorithms based on interpolation of the intensity or the correlation coefficient matrices.

The results of the correlation analysis algorithm are in-plane displacements described by a vector with the components  $D_x$  and  $D_y$  and the correlation coefficient  $K_{max}$ . Using a field of measuring points distributed in a defined way in-plane-displacement fields can be calculated. Often used types of grids are spatial ones or grids corresponding to a FEM-grid in order to compare the experimental results with numerical simulation. Assuming typical values of the field-of-view  $l_x, l_y = 5 \text{ mm}$ , the camera pixel amount  $n_x, n_y = 1024$  and the measurable subpixel shift  $p = 0,2$  (estimated), a measurement resolution of a single one point deformation measurement of  $1 \mu\text{m}$  can be obtained. Also 3D-deformation field measurement is possible by combining the digital image correlation analysis with stereo image acquisition and photogrammetric algorithms.

To study behaviour and parameters of materials and material combinations used in micro- and nanotechnologies, the method of grey scale correlation analysis has to be combined with various microscopic image acquisition techniques, e.g. optical microscopy with CCD-cameras, scanning electron microscopy, laser scanning microscopy, atomic force microscopy or micro tomography. Under the precondition of stable image acquisition conditions resolutions of some nanometers can be achieved.

Applications of grey scale correlation at digitised images in MST in the last decade of the past century have been established in R&D, e.g. to evaluate the reliability of components under mechanic or thermal load or to determine material parameters. Important future trends are seen in the field of industrial quality and production control of microsystems (especially in function test of mechanical or thermomechanical actors) and in growing activities in nanotechnology research («nanoDAC»).

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## Simulation of ferroelectric nonvolatile memory cells with MINIMOS-NT

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During recent years a special type of nonvolatile memory cells became more and more attractive, which takes advantage of the hysteresis properties of ferroelectric materials. To allow rigorous analysis of these devices several models were included into our simulator MINIMOS-NT, which allow a general transient two-dimensional simulation of arbitrary device structures.

MINIMOS-NT provides a rigorous approach to describe the static hysteresis properties of ferroelectric materials including the accurate modeling of subcycles [1] (Fig. 1). By now two different shape functions are implemented for the locus curves, based on  $\tanh$  and  $\arctan$ , respectively.

Two-dimensional simulation requires an algorithm that fulfills the geometrical and physical constraints especially in the context of field rotation. The algorithm implemented into MINIMOS-NT [2] is capable to serve this task both for isotropic and for anisotropic ferroelectric materials as well.

Increasing clock frequencies lead into a regime where the frequency dependence of basic material parameters like coercive voltage and remanent polarization can no longer be neglected. Simulation in the frequency regime would be numerically cheap, but leads to reduced capabilities in comparison with simulation in the time regime. Especially in the context of arbitrarily shaped signals and relaxation effects the rigorous approach of our simulator MINIMOS-NT is mandatory. By introducing three transient terms into the basic material equation, simulation of ferroelectric capacitors in a wide range of frequencies is now possible. Fig. 2 shows the simulation results for a capacitor

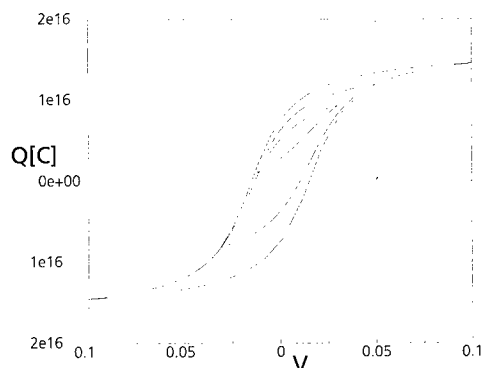


Figure 1: Simulated hysteresis including multiple subcycles

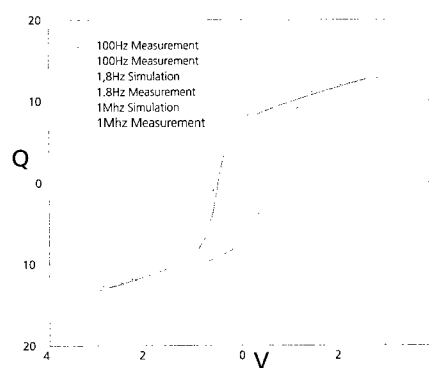


Figure 2: Q/V characteristics – Comparison simulation to measurement

and its agreement to measurements in a range beginning from 1Hz up to 1MHz. The application of the new simulation tool to circuit simulation is very promising. It can immediately be used for the extraction of specifications for the read and write cycles or the geometry of ferroelectric memory cells.

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## Thermo-mechanical reliability of microcomponents

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The computational design of reliable microcomponents as well as electronic packages can minimize expensive prototype development and testing. Accordingly, finite element (FE-) modeling is widely used to perform parametric studies on the thermomechanical behavior of electronic components like silicon microstructures, plastic packages, chip size/wafer level packages, or flip chip assemblies. From a mechanical point of view all these structures represent precision material compounds, which are subjected to different loading conditions. The theoretical analysis of stresses within these precision material compounds induced by environmental conditions require the characterization of loads and material properties, respectively, as well as appropriate failure criteria. Electronic materials with not very well-known mechanical characteristics have to be treated in the analyses. Frequently, the definition of loading conditions is not straightforward, because stresses can be induced during the processing history. The material compounds are usually stress free at one processing temperature, but chemical stress sources like e.g. migration, oxidation or polymerization can occur even at this temperature. These residual stresses are typically overlaid by thermal mismatch induced stresses during cooling from the processing temperature or during environmental temperature changes. Within electronic polymers, which are typically part of the material compounds, moisture swelling can induce subsequent stress states.

Constitutive properties of filled epoxy materials and soft solders are discussed. Investigations on many polymeric materials have shown dependencies of their mechanical characteristics on moisture, temperature and time. All tested commercially available encapsulants and underfill materials, most of them filled epoxies, exhibit a sharp drop in mechanical stiffness when their glass transition temperature  $T_g$  is reached, while the thermal expansion coefficient (TCE) strongly increases, as has been observed by DMA and TMA measurements. Additionally, the constitutive reaction of the filled polymers becomes time-dependent at this temperature level.

The paper provides an overview on those simulation related problems. It is shown that temperature and time dependence of the material characteristics as well as their non-linear character have to be considered in the analyses. Different examples demonstrate recent research activities in this field.

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## Analysis of an electronic structure bearing deep cracks

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Up to the electrical failure of an electronic component, as e.g. a flip-chip, a deep crack can grow into the structure. Up to this final failure a sequence of different failure steps occurs:

- A Manson/Coffin-type lifetime rule assesses the cycles up to the initiation of a macro-crack, that is the beginning of the loss of a structure, e.g. a bump, to bear the thermally induced mechanical load.
- Then, the macro-crack grows, but electrically the entire component can be still fully operating. This growing crack is running into a field of spatially changing stress, for example a field with a negative gradient of the stress intensity in the growth direction. But then, at a spatially decreasing stress intensity, the crack growth could be slowed down.

Thus, an *equivalent intensity of loading* for a region ahead of the crack tip should be used for lifetime assessment. Such an equivalent intensity could be simply a mean value of a local load (for example, the accumulated inelastic strain per cycle) taken over that region in front of the crack. By this driving force and a crack growth law of Paris-type, the number of cycles to tear this region can be given.

- Further, due to the growth of a large crack the geometry of the structure changes and thus, for example, the stress field changes and peak stresses can be reduced.

Therefore, the real lifetime of a component part can be much longer as assessed by a simple procedure using only the crack driving force quantity in the near of the point of the highest loading, i.e. at the location of crack initiation.

In order to take into account such effects as a **crack growth delay due to a gradient of the loading intensity** and a **partially unloading due to the growth of a large crack**, for a trustworthy lifetime assessment it seems to be necessary to calculate the correct loads within the cracked structures which arise in the course of the deep cracking. The electrical lifetime e.g. of a flip-chip without underfiller, could be reached when a certain internal bump, which is not subjected to the highest loading, is fully cracked.

*Cracked solder bumps* have been analysed by finite elements using a viscoplastic model presented at the MicroMat 1997. The equivalent load intensities for certain regions and the connected lifetimes, calculated within different configurations, cracked or not cracked, are compared with each other.

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## Scanning probe microscopy in semiconductor failure analysis

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About three years ago Scanning Probe Microscopy (SPM) has been introduced in the failure analysis department of Infineon technologies AG (former Siemens Semiconductor). Since then it has gained increasing importance both as a standard imaging tool for topography maps and even more so for specific measurements with derivative scanning probe methods.

One of the main reasons which led to the purchase of an Atomic Force Microscope (AFM) was the introduction of CMP in the Infineon fab in Munich. The CMP process had to be optimized and so the relation between the different process parameters and the resulting surface topography (»recess« and »dishing«) had to be investigated [1]. The need to obtain quantitative height information and the smallness of the variations of only a few nanometers made an AFM indispensable. By now AFM has acquired many more applications in semiconductor process control. It has a distinct advantage over other imaging techniques, like Scanning Electron Microscopy, whenever quantitative surface measurements are needed, when the variation are too small to be seen in SEM and when 3-dimensional information is required. So, e.g., etch processes in semiconductor fabrication and mask manufacturing are controlled by measuring step heights. New materials, like low-k, high-epsilon or ferroelectric dielectrics, are structurally characterized by their surface roughness. The 3-dimensional data allows critical surface features, e.g. divots at the edge or holes within a gate oxide, to be detected over an entire area and not only along a 2-dimensional cross section as in SEM and TEM.

Another important reason for the introduction of SPM in our failure analysis lab was the adaptability of SPM to various special measurement tasks. So the lack of methods to test thin tunneling oxides of EEPROM devices and MOS gate oxides for weak spots and homogeneity led to the development of a new SPM technique, Conducting-AFM. A voltage is applied to the bare oxide under investigation with a conducting probe tip and the resulting tunneling current is recorded at the same time as the topography. This method yields thickness maps of the oxide with Angstrom resolution in thickness and nanometer lateral resolution [2-7].

The measurement of doping distributions and profiles in semiconductor devices is another very important analysis topic for which SPM provides several solutions. As compared to established Secondary Ion Mass Spectroscopy (SIMS) measurements SPM techniques provide 2-dimensional imaging of dopant, or rather carrier, distributions on actual devices and not only depth profiles of large area test structures. Until now Scanning Capacitance Microscopy (SCM) measurements were carried out in collaboration with the University of Hamburg, Institute of Applied Physics, to solve daily failure analysis problems where misaligned or wrong implants were involved. The amount and importance of dopant mapping has led to the purchase of a new SCM machine which will shortly be installed in our lab. Additionally, there now is a Scanning Spreading

Resistance Microscope (SSRM) in the clean room of our Munich fab, mainly for the characterization of sub 0.1  $\mu\text{m}$  devices. To evaluate the benefits and drawbacks of both SCM and SSRM for practical dopant mapping in failure analysis and device characterization will be one goal of future experiments and theoretical calculations. Special attention is paid in this context to a quantification of these measurements to yield absolute carrier and doping concentrations and for the accurate delineation of pn-junctions.

Further SPM related investigations carried out by the University of Duisburg and the University of Wuppertal, in collaboration with and supported by our department, are the development of techniques to measure currents in operating ICs with a magnetic sensor attached to an AFM probe (Duisburg [8]) and to image temperature distributions with high accuracy for failure localization [9].

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## Material aspects for mass fabrication of microdevices by molding processes

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In order to utilize the potential of molding processes in mass fabrication of micro-devices a number of specific aspects have to be considered which deal with highly parallel batch type processes as well as the selection of corresponding functional materials and the final assembly and test procedures. A novel method for such batch fabrication of micro devices is based on the principle that a preform with a huge number of single microstructures is generated by means of injection molding, casting or embossing where each structure is firmly connected to a common sprue plate. The appropriate mold insert comprises a corresponding number of single mold cavities, each representing the complementary form of a microstructure and is realized e.g. by means of lithographic and electroforming, deep etching, photo ablation or ultra precision mechanical processes. The preformed microstructures are covered with a layer of a further material, e.g. by means of vacuum casting of a resin, so that a compound plate with embedded microstructures is obtained. By means of mechanical machining the common sprue plate and a part of the resin layer is removed which finally results in a resin layer with separated embedded microstructures.

Organic and anorganic polymers, reaction resins as well as ceramic and metallic micropowders can be processed in this way. Because each mold cavity is connected to the large cavity of the common sprue plate, a gate system with favorably low flow length inherently exists and even highly viscous materials can be applied in microinjection molding. In detail, engineering and standard plastics as well as metallic micropowders were analyzed successfully in a number of experiments, which demonstrates that a wide variety of materials can be applied for this highly parallel injection molding process.

A modified version of this process can be applied to generate a multiple template for electroforming processes which allows to fabricate directly microstructures from metals and metal alloys. In addition, thin films can be deposited very simply by means of CVD and PVD processes on the microstructures attached to the common sprue plate which e.g. serve the purpose to reduce friction or to increase surface hardness.

For a variety of microdevices it is of major interest to apply a combination of materials. This is not only feasible by means of additional thin film deposition or plasma treatment as mentioned above but also by means of combining an injection molding process with a subsequent embossing step which generates additional cavities to be filled with other materials. Corresponding examples deal with the insertion of metallic shafts into plastic gear wheels or dots from magnetic materials for position measurements.

A major advantage of the multiple injection process is based on the fact that all microstructures embedded in the second auxiliary material are arranged in an exactly defined position and orientation. As a result a magazine of microstructures exists where these structures can be simply pressed out of the embedding plate provided that there is no undercut in the structure. In addition, test structures can be generated in parallel to the other microstructures which may simplify process and quality control. A number of companies have meanwhile started projects to utilize this mass fabrication process for commercial microdevices.

## Development and application of ultra fine pitch solder paste for advanced microelectronic packaging

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State of the art electronic packaging concepts are connected with higher I/O numbers higher performance and smaller dimensions. Pitches down to 200  $\mu\text{m}$  certainly will be introduced to the market in the very next future. In this connection new requirements for the packaging technology arise. This holds especially for the soldering technology. The availability of high quality ultra fine pitch solder paste still is a question of concern.

The capability of an ultra fine pitch solder paste depends on different parameters. One is the quality of the solder powder, for instance the content of oxygen and the distribution of the particle size. Another problem is the flux composition. It is well known that the results of soldering strongly are influenced by the solder paste application. A method has been developed at Technical University Dresden for the objective estimation of the printing capability of a given paste. This method was used in order to improve the technological properties of an ultra fine pitch solder paste. In the corrosion of electronic components the flux residues are of great importance. This is a main problem by going to smaller pitches. For that reason scientists of the FNE are working on the development of the solder paste without corrosive residues in a joint project.

As a first step a product will be introduced to the market with a good printing behaviour in the ultra fine pitch range. The technological capability is demonstrated using a test layout. Results are given regarding the reliability of solder joints.

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## Nanotribological characterization of brittle surfaces

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Because of the technical advances in the microsystems technique the requirements on the used materials are getting rapidly higher. Thus, for many applications the optimization of the materials surface is very important. However, their mechanical characterization especially in respect of their tribological characterization is still dissatisfying.

The aim is to find out the fundamental frictional behavior of brittle surfaces by using the hitherto knowledge in mechanical characterization of brittle surfaces and selective experiments. An elementary way to simulate the multidisciplinary nature of the tribological behavior of a single asperity moving against a solid surface is the scratch test with sharp indenters of Vickers or Berkovich type. Here this test is used with normal loads  $F_N$  in the mN- $\mu$ N range to investigate the tribological parameters of the material surfaces in penetration depths in the nanometer region. The results show a strong deviation from the known Amontons's law, because of their strong dependence on the normal load and the contact area. Therefore a new method of data analysis was developed, which allows a full interpretation of the load dependence of the friction coefficient for both sliding and abrasive parts during the scratch process. Particular attention is also focused on the effects of adhesion forces between the moving pairs. By taking into consideration various theories about the adhesion of solids an estimation of its influence on nanoscratching tests can be given. Additionally the deformed surface inside and outside the contact area was investigated by atomic force microscopy (AFM) measurements. The results give interesting indications about plastical deformation like wall formation or sinking in effects on glasses as well as properties of coated systems.

## Mechanical characterisation and simulation of packaging polymer curing

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A severe obstacle for computational prototyping of electronic packages is the deficiency to be able to adequately investigate the processing induced stress fields during and after fabrication. Therefore, possible damages originating from the fabrication process and the residual stress field directly after fabrication cannot be adequately evaluated.

The residual stress field is mainly due to chemical shrinkage and simultaneous stiffness built-up in encapsulates during the curing process, and afterwards the cooling down phase. The levels of processing induced stresses can seriously influence the stress fields under operating conditions and thus affect the critical states of stress and deformation.

In a first attempt to investigate processing induced stress fields, a cure-dependent linear visco-elastic constitutive relation is assumed and implemented into the FEM-program MARC. This constitutive relation is based on a theory previously developed for curing polyester resins. Curing-time dependent visco-elastic parameters and initial strains are obtained through specially designed DMA measurements. Because of frequency limitations of the present test set-up, a model parameter investigation can only be performed on relatively slow curing encapsulate polymers. The capabilities of the developed model are demonstrated through a FEM simulation of the stress evolution in a flip-chip package during curing and subsequent thermal cycling. Development of a test set-up for parameter investigation for (quite) fast curing polymers is undergoing.

For the parameter identification process, up till now just one-directional measurement data were used. These originate from the in thickness direction of the coin shaped specimen being considered. By doing so, implicit assumptions on the deformation in the direction perpendicular to the measurement have been made. The validity of these assumptions could be questioned because of the limited constrained chemical shrinkage. For this reason model verification is performed by considering FEM-simulations of the curing process of the coin shaped specimen with its intermittent DMA loading. Moreover, a series of relaxation tests with a step-deformation applied at different curing times, were performed. Comparisons with FEM simulation results of these step-relaxation tests are discussed. Necessary updates of the curing-time dependent model parameters will be highlighted.



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## Sensors and smart electronics in harsh environment applications

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Harsh environment is produced by e.g. chemical aggression, operation temperatures beyond 150 °C, high pressure, and radioactivity. The application of the normally used semiconductor materials – silicon, gallium arsenide – as sensors or signal processors is impeded by some of their basic physical properties such as mechanical hardness, chemical solubility, band gap, and low lattice atom displacement energy. This paper deals with some ways out of the above problems.

The first approach is the replacement of silicon and gallium arsenide by wide bandgap materials such as silicon carbide, boron nitride, and diamond. The main intention of this measure is the operation in a higher temperature range. However, some of these materials are immune against chemical attacks or possess high displacement energies so that they are good candidates for devices in radioactive environments. Some of them also display high mechanical hardness. Though at first glance this way may appear obvious, it is faced with many technological obstacles unknown for the classical materials. These problems and some exemplary technical solutions towards a device will be discussed.

The second approach is solely restricted to temperature. The basic concepts are: (i) spreading of heat generated by a (point) source, (ii) transfer of the heat from the spreader through the carrier into a micro-cooling channel, (iii) transfer of the heat by a liquid driven by a micropump, (iv) transfer of the heat from the liquid through the carrier to a secondary cooler, and (v) annihilation of the heat by the secondary cooler. This concept is implemented in many variants on electronic systems. Examples shown are a laser diode bar, an FR4 epoxy printed circuit board with internal channels, an  $\text{Al}_2\text{O}_3$  containing processor board, a copper foil container for medium power devices, and a ceramic carrier for large power devices. For some of them, detailed simulation and performance data are given.

## Powder injection moulded aluminum nitride for packaging of mm-wave circuits

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Frontends of a millimetre wave application consist mainly of an antenna and a module. The later contains the RF-components, which have to be shielded from humidity by the module housing. Transceiver modules in modern radar sensors are designed to work at frequencies in the micro- and mm-wave range up to 100 GHz. The GaAs chips, which are required for rf frequency circuits lose large amounts of energy heat in many applications. This heat must be dissipated and drained. So the module housing additionally has to provide features for cooling and electromagnetic shielding of the components. The integration of electrical, mechanical and thermal functions in millimeter wave modules produced in large quantities requires the application of new materials and manufacturing processes. Materials for packaging such kind of modules must offer the following features: low weight, electrical insulation, high thermal conductivity, low thermal expansion and a high-precision workability at low-cost level.

In modern designs for millimetre wave frontends the distinction between antenna and module gets more and more lost. Hence housings have to fulfil additional mechanical functions. This leads to an increasing demand for manufacturing techniques allowing extremely complex geometries to be formed.

The paper shows an approach fulfilling these requirements by applying aluminum nitride formed by powder injection moulding (PIM). Examples of housings show the advantages of the technologies applied. PIM allows to form complex geometries from ceramics at low costs. In the PIM process a mixture from polymer and ceramic powder (feedstock) is casted by injection moulding under high pressure. After debinding (extraction of the polymer) and sintering a structural part is obtained which is 20% smaller than the green part.

The used powder injection moulding technique and the sintering process are explained as well as the metallisation and the bonding technique.

**Key figures:** packaging, millimeter wave module, antenna, heat sink, material development, metal injection moulding, aluminum nitride

## In situ measurements of deformations on microelectronic components by microscopic methods

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Under function conditions microelectronic and microtechnical components and assemblies will be influenced due to thermal and mechanical loading. The global and local deformations, which can occur under loading conditions, will influence the function and the lifetime of components or the assembly.

In order to obtain a thermo-mechanically optimised assembly knowledge of the material behaviour and geometric changes under loading conditions are essential.

A tool to measure the deformation behaviour of loaded microelectronic or microtechnical components and their interconnections is the microscopic observation.

Using in situ techniques the microscopic observation and the measurement of forces, lengths and temperatures can be performed simultaneously. The regions of local deformations and local material changes are documented in a series of microscopic images. The interpretation of the whole data from in situ experiments gives information on the properties of the materials, the interconnection, the loading conditions of the single components. Also reliability assessment and life time predictions can be derived from these investigations and compared with results of FE simulations [1].

The equipment developed for in situ measurements is a combination of a Laser Scanning Microscope and a micro deformation device »TMODE« [2] integrated in a heating chamber. With special imaging techniques and using the microDAC-measurement tool the fields of displacements due to loading conditions can be visualised.

At specimen of soldered and adhesive interconnections the sample preparation, the in situ measurement procedure and results to be obtained will be demonstrated.

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## Modeling of the deformation behavior of a flip chip underfill for finite element analyses

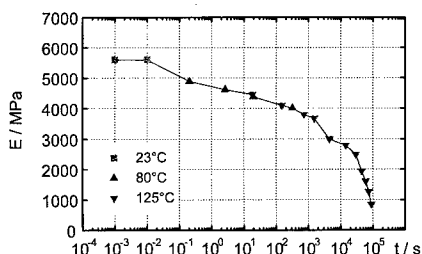
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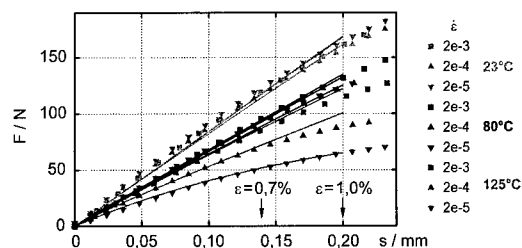
Finite element simulations have established as effective method for reliability assessment of electronic assemblies. The quality of the simulation results is significantly dependent on the applied material data. As the underfill layer plays an important role for the thermo-mechanical reliability of flip chip assemblies this paper focuses on the mechanical modeling of a representative underfill material.

The design of the experimental tests was adjusted to practical load conditions of a flip chip assembly. The measurement data showed that the underfill behavior is strongly temperature dependent and additionally time dependent at elevated temperatures. At strains larger than approx. 0.4%, the material behavior became stress dependent.

The processing of the measurement data aimed at the determination of parameters for Ansys' built-in material models. The extraction of parameters is completely introduced in the paper. A linear elastic model of the underfill described the mechanical behavior over a wide range of loads requiring very few parameters only. It offers precise solutions for a reduced temperature interval and is well-suited for trend analyses at optimized calculation times. A linear viscoelastic model considered time dependent properties and provided an exact representation of the underfill behavior (at the above mentioned small strains). It offers precise solutions for the complete temperature interval from  $-40^{\circ}\text{C}$  to  $+125^{\circ}\text{C}$ .



Master curve of the viscoelastic model



Tensile tests on underfill specimen: comparison of measurement and simulation

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## Influence of different modeling approaches for solder and underfill on the reliability assessment of flip chip assemblies

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Finite element simulations have established as effective method for reliability assessment of electronic products like flip chip assemblies. The quantitative simulation results are significantly dependent on the selected material models. Regarding flip chip assemblies, this statement mainly applies to the tin lead solder of the contacts and the encapsulant – the so called underfill. The availability of material data and appropriate modeling approaches are rather different for both materials.

Tin lead solder has been the object of studies for a longer time. Material data and model approaches are known in literature but most sources differ from each other. At TU Dresden, comprehensive studies on eutectic solder were performed and three modeling approaches were evaluated (target platform was the FEA code ANSYS):

- Anand's model (viscoplasticity)
- power law creep (with 2 terms) + plasticity
- sinh law creep + plasticity (as user defined model in ANSYS)

The availability of data of representative underfill materials is much poorer than compared with solder. Moreover, underfills are often modeled as very simple elastic materials. A dependence on temperature or deformation rate is mostly not considered. Three modeling approaches for underfill were evaluated:

- linear elasticity
- linear viscoelasticity
- Anand's model (viscoplasticity)

The properties of all above mentioned approaches are discussed in terms the simulated material behavior at various temperatures and deformation rates. For each combination of approaches, temperature cycling tests on a flip chip module were simulated by ANSYS. Different combinations of modeling approaches for solder and underfill led to different simulation results although each model was based on the same measurement data. The differences are discussed and conclusions are drawn which modeling approach is preferable for typical applications.

## Thermomicroscopy of materials and devices at nanometer dimensions

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Use of a resistive probe that is heated up with respect to a sample's temperature allows the determination of the locally existing thermal conductivity of materials. If this technique further utilises the 3w-frequency detection mode, a quantitative determination of the thermal conductivity can be gained which gives a material contrast imaging possibility with the scanning thermal microscope.

However, the quantification of contrast involves a frequency dependent calibration procedure that unavoidably is restricting the spatial resolution for quantitative measurements. This issue can be overcome by the introduction of a new developed resistive tip based rather on semiconductor properties than on the metal probes used up to now. The higher frequencies usable with this probe reduce the interaction volume between the sample and the thermal wave as generated by an AC driven thermal tip. The improved temporal behaviour of the new thermal probe is also leading to a reduction of the image acquisition time for temperature measurements with the scanning force microscope since the sensor is able to follow temperature changes at the sample surface even at higher scan speeds.

Aside of the higher spatial resolution of a microscopic thermal probe based on semiconducting materials an improvement of the temperature sensitivity can be achieved as a result of the exponential dependence of the electrical resistance of its temperature and due to the reduced volume of the probe fabricated with microstructure technology.

In this work quantitative conductivity measurements using the 3w technique on semiconducting materials were carried out, the improved performance of the new developed thermal probe will be demonstrated.

## Rapid Prototyping of components and demonstrators in microsystem technology

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Rapid prototyping is an innovative tool for the fast and low cost manufacturing of free shaped components. Rapid prototyping is especially effective in the production of special equipment, test components and small series. Applying different technologies, among them stereolithography (STL) and selective laser sintering (SLS), a wide range of applications can be covered in the automotive industry, medicine, and other industrial fields, particularly casting technology. Caused by the enhanced development of new systems which will allow the design of new structure generations with filigree geometry, rapid prototyping offers possibilities for new applications in micro technology and MEMS, too.

The rapid prototyping technology can be subdivided into the following basic elements:

- **Reverse Engineering:** *generation of 3D CAD data by scanning of real objects applying either optical or tactile measuring systems*
- **Virtual Prototyping:** *design of free formed 3D volume models using CAD-systems like I-DEAS, PRO-ENGINEER or CATIA*
- **Slicing of the Models:** *computer-based resolution of the object in defined layers (layer thickness of micro components  $\leq 0.05$  mm)*
- **Manufacturing of models by processing of the numerical data applying stereolithography and selective laser sintering**

These models can be duplicated by means of further technological steps

- duplication by plastic casting in a vacuum
- duplication by metal precision casting (casting material: titanium, stainless steel, aluminium, zinc, silver and others)

Essential process steps will be demonstrated for a fluidic-demonstrator developed for modular assembling of microsystems (MST-Baukasten) and other microcomponents.

## The thermo-mechanical analysis of twin-chips silicon piezoresistive pressure sensors

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The off-line optimization of silicon piezoresistive pressure sensors requires the detail knowledge on output voltage signal reaction not only to measured pressure but also to thermal stress. In this work the output voltage signal was numerically calculated using finite element method. However, such a numerical modeling of silicon piezoresistive pressure sensors usually is error biased mainly due to wrong assumptions. A wrong or uncertain material properties assumed as well as improper physical model and/or boundary conditions applied can cause the serious discrepancy with experimental results.

In this work we attempted to identify the significant source of errors in numerical calculations. Most often while performing numerical modeling of pressure sensors it is assumed that silicon has isotropic mechanical properties. In our calculations we assumed an anisotropic model. It was stated that calculated output voltage signal in the case of anisotropic model was considerably more accurate than in the case of isotropic model. In order to estimate the accuracy of calculations and correctness of assumptions taken into consideration the real sensors output voltage were measured and the results were compared with numerically calculated ones.

To avoid a large sensor voltage offset and its serious dependence on ambient temperature fluctuations the new method of silicon pressure sensor packaging in twin-chips configuration can be used. Instead of horizontal silicon-glass to substrate configuration the vertical configuration of twin-chips silicon-glass devices is recommended. Since the distribution of thermal stresses is in this configuration the same both for reference and measuring membrane, almost total reduction of thermally induced errors by subtracting sensors output voltage signals is expected.

Using above-mentioned method of numerical modeling we proved that a packaging of silicon piezoresistive pressure sensors in twin-chips configuration might become very attractive. We have stated that this method of packaging enables reasonable lowering of voltage offset and makes this voltage insensitive to ambient temperature fluctuations.



## Advanced measurement techniques for microstructured surfaces

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Micro structure technology (MST) has been developed to a full production technology in the past 5 years and made the step from pure research to real applications. The focus in the past was always to MST itself, how to generate devices and features in the respective small dimensions. Even though there was some attention to other subjects around the sole structures, like mass production, cost reduction and new applications, nobody really thought about the needs for building a supplier culture, like in »main stream« industries. But this is necessary to serve the future markets. Now, if discussion is going into supplier structures, the discussion about metrology is the next step. Some aspects of metrology needs in MST, which are definitely different to common production industry, are discussed in this talk. Also some new approaches for meeting these needs from the instrumentation side are given.

## Mechanical properties of electroplated nickel

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### Introduction

Micromechanical applications require materials with elastic behavior. Materials to fabricate elastic micro structures like accelerometer or micro-springs need to have high Young's modulus and elastic behavior over a wide mechanical strain range without plastic deformation.

Commercially available nickel electroplating electrolytes are usually optimized for surface properties such as brightness, wear resistance and corrosion behavior for coating purposes. In order to manufacture microparts electrolytes and process parameters must be optimized towards defined mechanical bulk properties to meet application specific requirements.

Plating parameters and electrolyte composition effect the solid-state properties of electroplated metals. Organic additives e.g. brighteners reduce grain size of metal depositions and introduce foreign atoms into deposited metal. Further influence to grain size can be obtained by current density. Grain size as well as texture effects have influence on elasticity and deformation characteristics of metals. Application of pulse plating is a promising technique to influence grain size as well as deposition thickness distribution.

### Tensile testing

The paper presents mechanical properties of electroplated nickel structures fabricated under varied deposition conditions. Samples for tensile tests were realized using UV-depth lithography and a nickel sulphamate based electroplating process (Nickelsulphamate EL, Enthone OMI-Blasberg). Fig. 1 shows the geometry of the samples for the micro tensile tester. The tensile tests provide information about mechanical properties of the deposited nickel samples. The experimental procedure is described in [1].

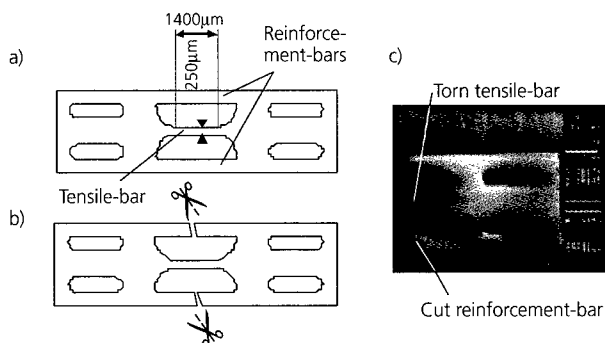


Figure 1:  
Samples for micro tensile tester:  
a) Geometry  
b) Reinforcement-bars are cut  
before testing  
c) SEM-micrograph of torn sample

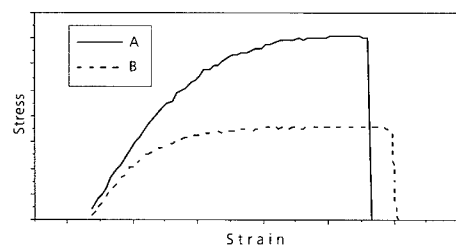


Figure 2:  
Stress-strain data of two different nickel samples

### Material characteristics

In fig. 2 stress - strain data of two differently processed nickel samples are depicted. Sample A was deposited from electrolyte with brightener additive (additive EL, Enthone OMI-Blasberg), whereas in sample B no brightener was added. Both samples were deposited under pulse plating condition at average current densities of  $15 \text{ A/cm}^2$ .

Fig. 2 clearly shows two different types of mechanical behavior. Sample A behaves less ductile and more brittle compared to sample B. The fundamentally different mechanical behavior is induced only by brightener additive.

X-ray diffractometry (XRD) Theta/2Theta-measurements (Bragg-Brentano geometry) of both samples are displayed in fig. 3. The XRD-plot of sample A shows broadened peaks. This indicates small grain size as can be found in nano-crystalline materials. The

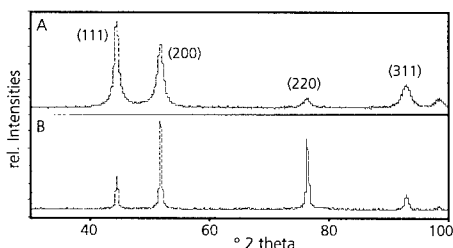


Figure 3:  
XRD measurement of nickel samples A and B

relative peak intensities show a nearly grey texture of anisotropic nickel. Sample B shows sharp peaks in XRD which are typical for larger grain. The texture of sample B differs compared to that of sample A. It shows an unsharp (220)-texture.

These first results show that nickel micro structures from the investigated electrolyte with brightener additive are elastic over a wider strain range than nickel from electrolyte without brightener.

A promising technique to determine Young's modulus with an high accuracy is characterization of micromechanical surface resonators. This concept and the corresponding results will be presented in the paper.

Elastic micro structures especially planar micro-springs are currently under investigation for application in IC-testing. To design and simulate micro-springs for this application reliable material data is of substantial importance [1][2].

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## Analysis of the injection level and temperature dependence of carrier lifetime by microwave photoconductivity decay

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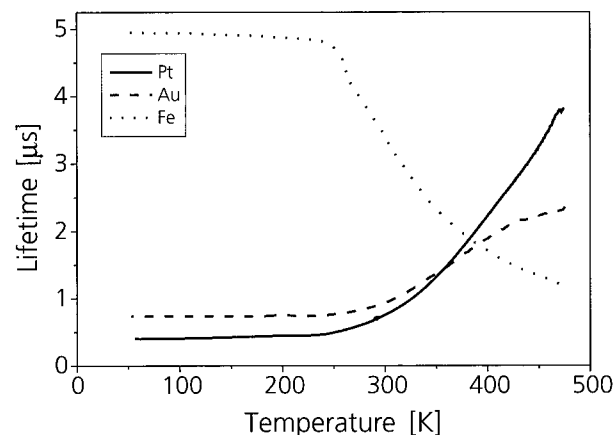
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Laser-induced and microwave-detected photoconductivity decay ( $\mu$ PCD) has proven to be an effective contactless and non-destructive measurement method for lifetime mapping on semiconductor wafers with a spatial resolution of up to 250  $\mu\text{m}$ . The routine application of this technique provides valuable information about the lateral distribution of contaminants down to concentrations of about  $10^9 \text{ cm}^{-3}$ . Even long lifetimes are measurable by applying a special chemical surface passivation to the non-oxidized wafers in order to reduce surface recombination. Furthermore, by variation of the laser power the injection dependence of lifetime can be determined at an arbitrary position on the sample under investigation. In addition, the dependence of lifetime on temperature can be analyzed using a suitable cryogenic apparatus.

With the refined variant of the  $\mu$ PCD technique we investigated the injection level dependence and the temperature dependence of the carrier lifetime in n-type silicon which was subjected to heavy metal doping. The samples were doped with platinum, gold, and iron, respectively, during crystal growth. The heavy metal concentrations were selected to be about a tenth of the phosphorus doping level, resulting in lifetimes of the order of 1  $\mu\text{s}$ . Under these conditions surface recombination can be ignored.

The carrier generation was varied over nearly three orders of magnitude around a relative injection level  $\delta n/n=1$  to cover the desired range of excess concentrations. We found a monotonous increase of lifetime in all three samples.



The temperature dependence of lifetime was analyzed in a temperature range from 50 K to 475 K. We observed similar behavior of the lifetime versus temperature relations of n-Si:Pt and n-Si:Au as displayed in the figure. The lifetime remains nearly constant at temperatures lower than about 230 K, then increases with increasing temperature. This behavior can be explained using standard

Shockley-Read-Hall recombination statistics if the individual recombination centers specific to the respective doping elements are properly taken into account. Comparing calculation and measurement we find that in n-Si:Pt the acceptor level of Pt dominates the lifetime in the lower and middle temperature range, whereas in the higher temperature range a contribution of the mid-gap level becomes relevant. In n-Si:Au the donor level mostly affects the lifetime at lower temperatures, while at higher temperatures the dependence is governed by the acceptor level. The lifetime of the iron-doped sample shows decreasing lifetime with increasing temperature at temperatures above about 250 K. This behavior can be explained by an exponential increase of the hole capture cross-section of the interstitial iron donor.

## Plastic reshaping of silicon microstructures: process, characterization and application

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The plastic deformation of silicon is possible at temperatures  $>650\text{ }^{\circ}\text{C}$ . By deforming silicon microstructures new shapes can be created which are not possible by using other techniques. Therefore new applications become possible, for example as

- components which are partially situated outside the wafer plane
- components that exert an elastic force on others as a result of the assembling
- components with a changed slope of definite crystal planes.

For the presented studies silicon microstructures like beams fastened at one or two sides, membranes and angled stripes were integrated into a wafer, prepared by wet anisotropic etching (KOH) and then plastically deformed [1]:

The reshaping process was performed in different ways:

1. Deformation with tools inside a testing machine equipped with a furnace (homogeneous heating of the whole element)
  - deformation of single structures with a silica rod or special reshaping tools (stamp and rest also fabricated from silicon),
  - »batch process«: simultaneous deformation of all elements inside the wafer with reshaping tools (stamp and rest wafer out of silicon).
2. Deformation by laser beaming without tools.

Using the silicon tools very exact formed shapes can be reached. The bend is limited by the depth/height of the tools. By laser beaming it is possible to deform the microstructures without contact up to angles of about  $90^{\circ}$ . With this method elements can be plastically deformed that cannot be completely heated, too.

For the plastic deformation of dislocation free monocrystalline silicon the generation of dislocations is necessary. The created dislocation structure was analyzed by the etch pit technique [2]. The zone of generation of the dislocations is of special importance for smallest bendings of the silicon microstructures. By the method of IR microscopy decorated dislocations in this area could be made visible. The first dislocations are generated shortly before reaching the upper yield point. After crossing this point the density of dislocation increases.

The introduced dislocations during the plastic deformation influence the mechanical properties of silicon at room temperature. These properties are of interest in view of the application of the microstructures and therefore must be known. The attention was first drawn to the static strength of the elements. As a basis for this the fracture strength of deformed and undeformed silicon beams (prepared by wet anisotropic

etching in KOH) were measured in dependence of the deformation parameters (temperature, bending rate and bend), sample orientation, sample geometry and surface quality. The measurements were performed in 3-point-bending tests inside a DMA. The fracture strength of undeformed polished beams (250 MPa) is in good agreement with the values of other authors. After a previous deformation with homogeneous heating the fracture strength increases. First measurements concerning the fracture strength of undeformed and deformed microstructures were carried out.

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## Characterization of sol-gel derived films in mesoporous matrices

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In this paper I would like to summarize our results on Er and Tb doped sol-gel derived films fabricated on mesoporous silicon and anodic alumina. The samples were characterized by the means of SIMS, SNMS, RBS, TEM and SEM analyses evidencing fabrication of silica and titania xerogel films on the wall of a mesopore in a layer several microns thick. Strong  $\text{Er}^{3+}$  and  $\text{Tb}^{3+}$  luminescence from the xerogel films with predominant bands at 1.53  $\mu\text{m}$  and 454 nm, respectively, has been observed from the fabricated samples and investigated in a temperature range from 4.2 to 300 K. Some technological factors towards enhancement the luminescence of lanthanides from the xerogel films will be discussed.



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## Development of lubricious DLC coatings for microsystems

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The DLC (Diamond-like Carbon) coatings have remarkable tribological properties due mainly to their good excellent frictional behaviour. These coatings can be applied in many industrial applications where sliding or contact generates wear and high frictional forces on the components.

This work reports on the development and tribological characterisation of functionally gradient DLC coatings. The PVD-Magnetron Sputtering technique has been used as deposition method, using metal interlayer in order to improve the adhesion of the coating to the substrate. Furthermore, the coatings have been built with a gradual change in the composition to decrease the internal stress and to avoid delamination.

The quality of the films has been examined by scanning electron microscopy and Auger electron spectroscopy and the tribological characterisation of the DLC coatings has included frictional tests using the ball-on-disc geometry, leading to friction coefficient values below 0.2 with no measurable wear on the surface. Besides that, microhardness and elastic properties have been measured by a dynamic microindentation technique and adhesion of the coatings to the substrate has been evaluated by the scratch test method.

## Dicing challenges in microelectronics and micro electro-mechanical systems (MEMS)

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### 1 Introduction

Both in semiconductor and micro electro-mechanical systems (MEMS) technology, wafer fabrication batch techniques are used to build up microdevices. A back end machining process is detaching the individual components by separating the wafers into chips. While the semiconductor industry mainly uses silicon as wafer material, in MEMS ceramics or glass is employed also. Both groups of materials exhibit a brittle dicing behavior. Brittle mode dicing results in microcracks being induced in the sidewalls as well as in chipping at the edges. While minimizing the chipping has been a priority back end fabrication issue for quite some time, sidewall microcracks so far have been ignored, although obviously these micro cracks may be a potential starting point for a fracture. However, new packaging techniques like flip-chip are increasing the thermal strain while growing safety demands put new emphasis on chip reliability. This paper presents an approach to dicing resulting in minimizing the inducing of microcracks as well as lowering chipping.

### 2 Ductile Machining of Silicon and Ceramic Wafers

Typically, when dicing brittle materials like silicon or ceramics a brittle mode cutting takes place. In this mode, during cutting a dicing wheel's diamond grain engaging at the wafer induces microcracks in the material. As a result, a network of microcracks extends far below the surface created by machining. As a result of brittle mode cutting the material suffered substantial subsurface damage.

As an alternative, by choosing the appropriate cutting parameters, even brittle materials may be machined by ductile mode cutting. Typically, by selecting a suitable cutting energy as well as an individual depth of cut in the order of a couple of hundred nanometers, ductile mode cutting may be achieved.

### 3 Blade Alternatives

For cutting semiconductor materials or ceramics, diamond wheels consisting of diamond grit matrix in a binder are used. Best cutting results are achieved with resin bonded wheels. The diamond grain holding force of the resin binder is small, therefore any diamond grain dulled due to wear brakes lose. Since the binder is soft, the wheel suffers circumferential wear, thus allowing virgin diamonds to engage in the cutting. While the cutting performance of a properly selected resin blade is excellent, its wear however is high. Therefore, despite its good performance, production usually shies away from using resin blades.

#### **4 Production Type Ductile Mode Cutting**

In production metal bonded wheels typically are preferred. This type of wheel has a much greater diamond holding force and therefore wears much slower. It also can be used at much higher feed rates than a resin bonded wheel. However, the diamond wear causes a duller wheel. It is more difficult to keep it sharpened, a key condition for achieving ductile mode cutting.

One of the highest quantity MEMS type applications are thin film recording heads for rigid disk drives. In 1999, more than one billion parts were fabricated. The wafer is made out of alumina-titanium carbide (Altic). Rather than being merely diced, there are squareness and flatness requirements for the parts' sidewalls to be taken into account since they are serving as tooling reference surfaces. Ductile mode dicing was considered highly desirable for this application due to it resulting in an optimal surface finish and minimal chipping. The approach chosen was to find a compromise between the cutting performance of resin and metal bonded wheels. First, a soft metallic binder was chosen and second, a dressing stick was used for in process dressing to keep the wheel sharp.

Such an approach is also very promising when implementing silicon machining. It is opening a possibility to minimize subsurface damages and reduces the risk of fracture initiation.

#### **5 Conclusion**

For many applications achieving the highest possible cutting speeds will be considered the ultimate process criteria. However, such an approach results in brittle mode cutting of the wafer, causing substantial subsurface damages by inducing microcracks. These microcracks may make it much easier for the chip to fracture when exposed to dynamic thermal strain. Ductile mode cutting, on the other hand, opens up a path to dice chips close to subsurface free of damage, thus removing this liability. By carefully choosing an appropriate wheel, the compromises to be made in regard to cutting speed are tolerable.

## Gradient-enhanced macroscopic constitutive principles for the mechanical description of microstructures

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The macroscopic modelling of microstructural phenomena in many materials has been the subject of intensive research in the past decade. Gradient-enhanced macroscopic continua are typical examples, which were mostly used to construct a macroscopically sound model of irreversible processes in a material. Various nonlocal or gradient-enhanced damage models [2] and gradient plasticity models [1] have been developed, each with its own assumptions and limitations. Although these higher-order continuum models are well motivated from a computational point of view, the physical relation with the micromechanics remains vague. The only microstructural parameter that appears in these models is an intrinsic length scale, which is assumed to be related to the microstructural geometry and the occurring failure mechanisms (decohesion, void growth, debonding, granular deformations, intergranular cleavage, crystallographic slip, dislocation dynamics, etc.).

In order to analyse the physical implications of a specific choice of a macroscopic gradient enhancement, the analysis of the phenomena on a smaller scale which is representative for the microstructure is appealing. Micro-macro homogenization analyses are commonly used to obtain a macroscopic response that matches the influence of the microstructural events as good as possible. One class of homogenization techniques still uses a closed-form macroscopic constitutive relation, while another class evaluates the macroscopic stresses in each material point directly from the response of a microscopic representative volume element (RVE, multi-level techniques). Assumptions have to be made in these homogenization strategies, which again will have an influence on the macroscopic constitutive response that is generated.

It is clear that assumptions are being made in both macroscopic gradient theories and microstructural RVE approaches. These assumptions inevitably lead to limitations, which are not well understood so far. Connections between gradient theories and micro-macro models are still rare. This contribution focuses on this relation and tries to establish links between certain classes of gradient models and the associated deformation modes on a microstructural level. Attention is given to explicit and implicit gradient enhancements, nonlocal strategies, where higher-order terms can be used in either the deformation modes and the resulting Cauchy stresses, or even both (e.g. mechanism-based strain gradient theories).

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## Dielectric strength of ceramic substrates

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Ceramic, glass-ceramic and glass substrates are important structural components for the packaging of microsystems. The trend of microsystem constructions shows the development to high performance, high reliability and low cost products. Important features of this development are i.e.:

- a zero defect philosophy of circuits operation also under extreme climate conditions
- hermeticity of dielectric structures (high density, low porosity),
- excellent electrical insulation in a wide range of frequencies,
- miniaturization of circuit size and a greater circuit interconnection density.

This requires the testing and process controlling of the different substrate properties, including the testing of the dielectric strength. Especially the measurements of the dielectric strength have to be carried out under comparable conditions. The test conditions have to be known for the evaluation and the comparison of the values of different materials. The dielectric strength of insulating materials is the voltage gradient at which dielectric failure of the insulating materials occurs under specific conditions of the test (electric breakdown voltage).

A computer controlled high voltage laboratory equipment WGBS 4,4/35-50 HPS (High-volt Dresden GmbH) was used to measure the dielectric strength of thin high alumina ceramic substrates under different test conditions. The use of maximal voltage AC 35 kV or DC 50 kV, different shapes and sizes of the electrodes and different rates of increasing voltage were investigated.

As fired substrates of different thickness and substrates reduced in thickness by grinding were measured under the same test conditions to investigate the dependence of the measured values from the sample thickness.

The results are discussed taking into account the influence of the microstructure (pore sizes distribution, grain sizes) of the examined substrate samples and their surface quality (roughness, waveness).

## Development of contact and wiring systems for high temperature sensor applications

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Sensors for high temperature applications are subject matter of recent and current research activities. In future the demand for such devices will be dramatically increased. Possible application fields are the environmental technique (gas control systems), the automotive industry (combustion and engine control) as well as space and aircraft industry.

These applications require temperature stable device concepts, materials as well as contact and wiring systems. Even at temperatures above 200 °C various interactions (i.e. solid state reactions and diffusion) could start or will be accelerated (see Figure 1). This means the contacts, interconnects, and connections to the next wiring level must be physically and chemically stable in a wide temperature range during the whole device life time. As an additional difficulty the uppermost metallization layer (bond pads) as well as the bond wire is often exposed to non inert environments, i.e. air. So the here used materials should not react with oxygen. Otherwise a protection against oxidation and corrosion is necessary. Regarding this requirements only few materials could be used for the metallization in high temperature devices. A further demand to the chosen materials is the compatibility to the microelectronic technology.

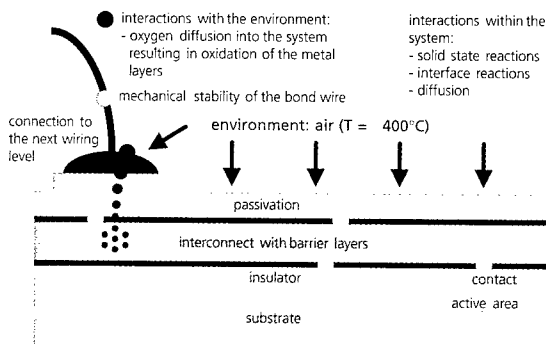


Figure 1:  
possible interactions in a temperature loaded contact and wiring system

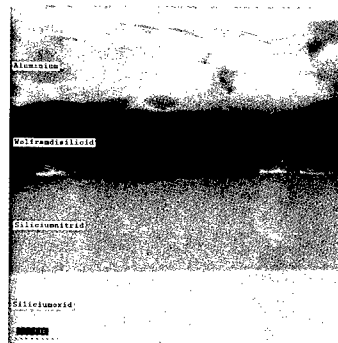


Figure 2:  
cross section of the Al-WSi<sub>2</sub> contact system after long term storage (2000 h) at 400°C

Within this work temperature stable contact and wiring systems for two sensor applications will be presented. The first application is a pressure sensor based on silicon carbide (SiC), which is able to monitor the pressure in a combustion engine. Temperatures up to 400 °C are possible for this sensor. For both, the ohmic contact to the SiC-substrate and the on-chip interconnects the used material is tungsten silicide ( $\text{WSi}_2$ ). Aluminium is used as bond metallization. The connections to the next wiring level were realised by aluminium heavy wire bonding. The stability of this system was proofed by analytical investigations (AES, X-TEM, REM) as well as contact and interconnect resistance measurements. No significant changes were found by these investigations after a thermal storage for 2000 hours at 400 °C (Figure 2). A sufficient mechanical stability of the wire bonds was found by pull and shear tests.

The second application is a gas sensor, which is able to analyse car exhaust fumes directly inside the exhaust. Here temperatures up to 500 °C were established. Titaniumoxide ( $\text{TiO}_2$ ) serves as sensitive layer because of the known thermal stability and the given sensitivity for the interesting gases. Platinum was chosen as material for the contacts, interconnects and bond pads. The outer electrical contact was realised by ultrasonic wire bonding using gold and platinum wire. With platinum wire first bonds could be realised, however the reproducibility up to now is not satisfactory. In opportunity to this results gold wire delivers stable bonds without any problems concerning reproducibility. The thermal stability of this system was found to be sufficient. For the examination analytical investigations as well as electrical measurements were applied.



## Thermal-fatigue kinetics in SMC solder joints by SEM/EDS fractography

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We submit evaluation of thermal fatigue (TF) reliability of SMC solder joints (SJ) by high resolution SEM/EDS fractography technique. This technique is shown to be in particular efficient in the observation of the early stage of TF damages - which is missed by traditional surface inspection techniques - and in revealing the weak interface links in the joint microstructures.

SEM/EDS inspection has been performed after standard accelerated thermocycling (ATC) of Cu/63Sn37Pb/FR-4 joints followed by pull-out test to expose the fracture surface which contains (i) fatigue striations zone and (ii) the dimpled zone of the overload ductile failure. It is shown that in the joints under investigation:

- The normalized surface area FF of the fatigue cracks which extend in the solder parallel to the lead-solder joint and very close to the intermetallic/solder interface grows exponentially with the number N of thermocycles  $[0-100]^\circ\text{C}$ , one cycle per hour in the range  $N \leq 2674$ . The average crack velocity is estimated as 0.15 mm/cycle.
- At  $N = 4300$ , 90 % of the joints are expected to have FF between 70 % and 20 % with the average fraction of the damaged interface  $FF = 40$  %.
- TF threshold falls in the range  $0 < N_{TH} < (600-800)$ , and is likely closer to the lower boundary of the interval.
- Although fatigue crack formation were observed in all parts of the joints, from 50 % to 100 % of the cracks form near the toe of the joints. This points likely to adverse wetting conditions and »a notch effect« caused by damage to the toe region from the cutting of the Cu lead.
- Up to 40 % of all thermal fatigue cracks form close to voids, in particular to larger voids which diameter exceeds 30  $\mu\text{m}$ ; these large voids are shown to originate from the entrapment of the flux in the joint and its outgassing in the sandwiched solder during reflow and concentrate mainly along the Cu lead-solder interface.

It is concluded that amassment of the FF(N) SEM/EDS fractography data for various SMC solder joints under ATC conditions will create a good basis for unbiased comparative assessment of their reliability and soldering technology beyond traditional inspection routines and standards.

## On the origin of the short line effect in electromigration

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Since its discovery by Blech, the short-stripe effect (SSE) has become a core element in the analysis of electromigration (EM) in thin film microelectronic interconnects. We show however that the classical Blech description does not fit accurately either the EM kinetics or the threshold data in short, unpassivated Al and Cu conductors.

To account for the EM behavior of short lines we develop further the theory of creep affected EM, with diffusional creep as a stress relaxation mechanism (*E. Glickman et al. JAP, 83, 100 (1998)*) for the near-threshold limit. Complemented by the idea that the conventionally measured EM threshold length ( $L_{th}^*$ ) is an apparent, kinetically affected, and not a true ( $L_{th}$ ) value, this approach - which emphasizes the role of a dynamic (adjusted to stressing conditions) EM stress - provides a self consistent, quantitative description of the EM kinetics and thresholds.

It suggests that *the diffusional creep viscosity  $\eta$  is the major material property which determines SSE in short interconnects*. The analysis extracts from experiments typical  $\eta$  and  $L_{th}$  values, which indicate that SSE clearly manifests itself even when  $L_{th}^*$  tends to zero. We submit that short conductors with  $L < L_{th}^*$  are generally not immune against EM contact openings, but migrate in a pure creep-controlled EM regime, in which *the overall EM rate is totally controlled by stress relaxation via creep into hillocks*.

In other words, the EM phenomenon which is considered conventionally as the »electrical« failure represents - for short, near-threshold lines - a »mechanical« failure. Considered from a wider perspective, this behavior is caused by transition from diffusion-controlled to the interface reaction (creep) - controlled regime of EM. Transition of this kind is the characteristic feature of diffusional mass transfer in small macroscopic and microstructure dimensions.

## CVD diamond and its application in medical technologies

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Diamond possesses outstanding material properties. It is chemically inert and therefore biocompatible. Diamond can be insulating and, if doped, also semiconducting or metal-like. It has a high mechanical strength and the highest thermal conductivity of all materials. Furthermore, diamond is non magnetic and therefore suitable for visualization in Magnetic Resonance Tomography (MRT). Because of the lack of reproducible fabrication methods, these outstanding diamond properties could not be applied in the medical field so far. Only the hardness of natural diamond stones is used in ultrasharp scalpels for ophthalmo- [1] and neuro-surgery. In the past 5 years, novel CVD-processes for diamond growth [2] and structuring have been developed. Using these processes, diamond films can be deposited homogeneously on silicon substrates. Furthermore, the operation of diamond microdevices such as transistors, diodes, sensors and actuators have been demonstrated successfully on these diamond substrates [3]. In the technical field, diamond already serves as a multifunctional material. Especially in the medical field, diamond may serve as the ideal material for the fabrication of multifunctional surgery tools. Here, the high mechanical strength of diamond enhances the approach towards further miniaturization of tools for minimally or micro-invasive surgery. Also the combination of cutting tools with sensors or actuators seems feasible now. Since the fabrication methods of these diamond tools are almost equivalent to

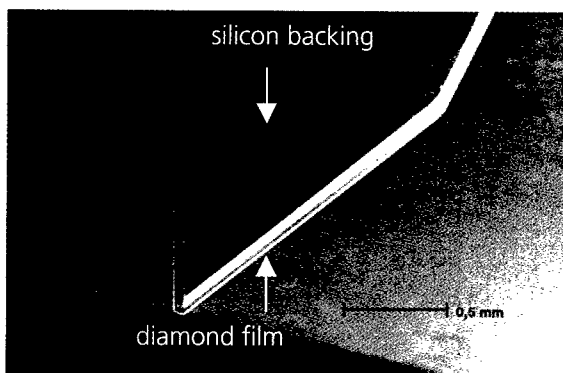


Figure 1:  
SEM-photograph of a diamond scalpel  
blade fabricated by microsystem  
technologies

that of the silicon technology, the size of these elements can be extremely small, even below 10µm. An overview, demonstrating the potential of micromachined diamond tools in medical applications will be given. Fig. 1 shows a SEM-photograph of a diamond blade used in ophthalmic surgery.

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## Analysis of structure and dimension of electronic parts with high resolution X-ray tomography

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Nondestructive Evaluation of electronic parts and components is often the only tool to characterize and analyze structures because a destructive method influences the defect itself. 3D - high resolution X-ray tomography offers the possibility to detect small structures and defects together with a dimensional analysis even of complex inner and outer geometries.

Equipment and methods developed at BAM to perform high resolution X-ray tomography were applied to a very broad spectrum of samples and test components. The maximum spatial resolution was improved to 2.5 micrometers using a transmission target X-ray tube together with a combination of a GdOS covered magnifying fibre optic taper and a CCD camera.

The reached spatial resolution opens new outlooks to characterize technical layers with a thickness of about 30  $\mu\text{m}$  and more. Results obtained with high resolution CT will be presented and discussed to demonstrate the wide range of applications of 3D-high resolution CT:

- structure and defects in glass fibre cables
- dimensional check of plugs and annexed cables
- performance test of springs in plug-ins
- characterization of technical layers

## Reliability risk assessment of organic flip chip pin grid array package under thermal and mechanical loading conditions

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Controlled collapse chip connection (C4) or flip-chip, a concept that is originally devised at IBM, has become the emerging interconnect technology for high performance microelectronics. With increasing demands on miniaturization, socketability, and cost reduction of future microelectronics, organic flip chip pin grid array (FC-BGA) has become a very attractive packaging solution. A FC-PPGA package consists of the solder interconnection of an integrated circuit (IC) directly to a low cost organic carrier with pin grid array solution. As such, in addition to providing higher density of input-output connection per chip area, FC-PPGA also offers a socketable packaging solution, something which cannot be achieved by its plastic ball grid arrays counterparts. Although the FC-PPGA technology renders definite advantages over the traditional surface-mount technology components, it also raises other reliability concerns. Excessive forces placed over the exposed silicon die surface during component testing or heat-sink attachment can cause package to flexure which ultimately can lead to severe damage of die and package. A comprehensive investigation has been conducted to determine the maximum loading limit of FC-PGA. A material testing system has been utilized to simulate various loading conditions (ranging from 15lbf to 200lbf) during heat sink attachment. The effects of permanent loading coupled with thermal cyclic loading to the overall reliability of FC-PGA structure are also investigated in this study. Three-dimensional nonlinear finite element models of FC-PGA package has been employed to aid in understanding the mechanical behavior of silicon die and organic carrier under mechanical and thermal loading conditions. Shadow Moiré technique has been used to measure the out-of-plane residual deformation of the FC-PGA components after undergoing simulated loading conditions. These components were then visually inspected and tested functionally. Results were summarized and used as indicator of package reliability.

**Keywords:** Flip Chip, Deformation, Thermal and Mechanical Loading Conditions.

## Modeling and testing of strength and fracture of microelectronic materials and components

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Strength, fracture and reliability of microelectronic components are strongly determined by essential difference of mechanical and physicochemical properties and microstructure of electronic materials, specificity of the electronic component geometry and complex nature of external actions (thermo-electromechanical loading, action of aggressive media).

Traditional models and methods for testing and evaluation of structural strength and fracture need to be adopted and modified for microelectronic components since these components and their elements have, in particular, as a rule such a combination of the geometric parameters which we do not observe in mashinary structures. For instance, the ratio of the metallization thickness to its length is rather similar to the one for the ice cover than to the geometric characteristics of usual structures.

The paper is devoted to an analysis and review of the characteristic fracture processes of microelectronic materials, elements and components, their modeling and testing. Special attantion is paid to the processes of metallization fracture under stress and electromigration and package fracture under thermomechanical loading and moisture influence. Kinetic processes of the adhesion fracture at the interfaces inherent to the components are discussed. Material and interface parameters which need to be measured or evaluated for prediction of the adhesion strength and fracture are considered. Results of the modeling of the adhesion defects nucleation and growth are presented. The relative influence of the mechanical, thermal and chemical loads is analyzed.

The system of tests usually used for an evaluation of the quality, lifetime and reliability of microelectronic components is discussed from the point of view of its necessity and sufficiency. Examples are given which demonstrate that often the test parameters do not provide a conservative estimate of the fracture conditions while in other cases they lead to too conservative estimates.

## Strain measurement in solder interconnects of advanced electronics packages

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Improvement of thermo-mechanical reliability of advanced area array packages depends by a large extend on the understanding of typical failure behavior and failure mechanisms. For that purpose direct strain and stress measurements on loaded test specimens are desirable. The authors made use of the microDAC deformation measurement technique to determine strain fields on thermally loaded, cross sectioned FC and CSP specimens. The method allows to resolve strain fields inside tiny structures like e.g. solder interconnects or conductive adhesive layers. They can be utilized to judge stress/strain situations on real components and to void severe stress concentrations, but also to verify modeling of finite element simulations. Early defect appearance and development can be observed because of displacement discontinuities along cracks or delaminations under load.

The paper presents results of strain analysis for different flip chip configurations and chip scale package types. Flip chip assemblies with eutectic PbSn solder bumps as well as ACA bonded dies have been investigated. It has been established that the major strain direction in flip chip joints is perpendicular to the die plane. Global shear of outward bumps is almost completely suppressed in most of cases by underfilling or by the adhesive layer. Stress compensation commonly arises out of package warpage. Furthermore, bump deformation can be strongly influenced by the glass fabrics of organic laminates used as board materials.

A main demand to chip scale package reliability is the avoidance of too large solder ball strains, which lead to material fatigue. Because CSP have to be assembled without underfilling, stress compensation mechanisms must be introduced to relieve CSP solder balls from severe loads. Different packages with rigid and flex interposers tackle the stress compensation problem in a different way. A first attempt is made to compare some of them basing on experimental strain and warpage measurements.



## Spectroscopic ellipsometry and dark conductivity measurements on p- and n-type microcrystalline silicon films

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Hydrogenated microcrystalline silicon ( $\mu\text{c-Si:H}$ ) deposited by low temperature plasma methods appears to be a promising material for photovoltaic and opto-electronic applications due to its properties such as high electrical conductivity, high optical bandgap, good transparency. A further advantage is the compatibility with a-Si:H deposition technology. To test  $\mu\text{c-Si:H}$  as emitter layer or backsurface field layer in heterojunction solar cells we deposited p- and n-type  $\mu\text{c-Si:H}$  thin films on Corning 7059 glass using the very high frequency glow discharge technique (VHF-GD, 110 MHz). For the n-type  $\mu\text{c-Si:H}$  layers we varied the main process parameters such as deposition temperature, process pressure, gas flow, doping; for p-type  $\mu\text{c-Si:H}$  layers, we varied the deposition temperature over a wide range (400 °C – 220 °C). The layers were investigated by optical and electrical measurements. The quality of the films has been evaluated by variable angle spectroscopic ellipsometry and electrical dark conductivity measurements using four point probe measurements at room temperature. To extract physical information (layer thickness, optical constants, surface roughness) from the measured ellipsometry data, an analysis based on different models was performed. In a first step, we used a semi-empirical single parameter dispersion model for polysilicon by W. A. McGahan [1] to characterize the  $\mu\text{c-Si:H}$  layer. In a refinement step, a parametric semiconductor model [2] was used. For all samples, the absorption coefficient is several orders of magnitude larger than that of crystalline silicon but lower than for amorphous silicon.

[1] W. A. McGahan et al., Proc. SPIE, 2725, pp. 450-459 (1996)

[2] Guide to using WVASE32™, J. A. Woollam Co., Inc. (1995)

## Atomic diffusion and phase stability in thin films

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The emerging importance of micro- and nano-scale devices, together with established areas such as integrated-circuit manufacture, bring relevance to studies of atomic diffusion and phase stability over short length scales. These thin-film studies can be particularly useful when carried out on multilayered samples. This presentation will review such studies, attempting to identify key issues for devices. One of the reasons for interest is that the reliability of devices may be adversely affected by changes in phase and microstructure of the materials present. However, it needs to be recognised also that such changes can be beneficial and may provide opportunities for tailoring properties and optimising device performance. And there are questions of fundamental interest in connection with changes of basic behaviour at short length scales.

Atomic diffusion and phase changes are likely to be accelerated in thin-film devices by the short diffusion distances. In addition, the possible high density of interfaces between reactive materials can provide a strong driving force for transformation. The diffusion and transformation phenomena in thin films are sometimes what would be expected on extrapolating bulk behaviour down to shorter length scales, but sometimes quite new forms of behaviour emerge. The implications for fundamental studies of diffusion as well as for properties (particularly magnetic) will be considered.

The presentation will focus on: (i) gradient-energy effects, in which the sign of interdiffusivities can be reversed; (ii) stress effects, which can invalidate the usual Darken analysis of interdiffusivity; (iii) phase nucleation and growth in thin-film reactions; and (iv) the stability of layer structure in otherwise stable, immiscible systems. Gradient-energy effects are significant on length scales of a few nm, but stress effects are much longer range, dominating at length scales less than about 20 nm. Phase nucleation and growth in thin-film reactions can be very different from bulk systems and progress in analysing the distinct behaviour will be reviewed. Finally, the study of immiscible systems is of increasing importance and recent advances will be outlined.

## Evaluation and testing of microsystems using optical methods

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It is increasingly important to apply nondestructive inspection methods to the fabrication of micro components and micro systems. The determination of these components during and after the application process reveals valuable information about the true function and the true working parameters. In this paper, Si-packages and 3D-microstructures produced by additive technologies are investigated in its process levels.

Optical methods, like the autofocus measurement technique and the phase-shifting interference microscopic technique fulfill the high demands of measurement techniques for the analysis of the samples. The autofocus measurement technique, for example, offers the possibility to analyze large lateral expansions of up to 200 mm x 200 mm and structural heights of up to 1 mm with a z-resolution of 0,01 per cent of the structural height. The surface of the object is scanned in a manner that can be defined for every object individually. The phase-shifting interference microscope optically reproduces the surface of the object. The microscope utilizes a special imaging measurement technique. The information on the 3D-geometry is available for every pixel of the shown surface. Structural heights of up to 100 µm can be examined with an accuracy in the range of Angstrom.

Furthermore a combination of thermal and optical analyses is used to determine the material characteristics of the components. Equipment was fabricated for the application of well defined thermal loads to the specimens and observe them by means of optical analyzing methods.

The applied 3D-microstructures are based on a Si-wafer as a substrate. To achieve even layers it is presumed that the wafer has no warpage. Considering the different materials on the wafer, an appearance of warpage is possible during the process. In this paper, different layers were examined on their process-temperature behavior.

To examine micro components in process it is necessary to keep them free of contamination. Therefore an in-situ measurement module is designed at the Fraunhofer Institute for Reliability and Microintegration. It includes several optical measurement techniques. The module has the advantage that it is not necessary to interrupt the vacuum regime and the cleanroom classification for the measurement. Optical measurements of Si-Packages within airpressure-dependent encapsulated vacuums were carried out. On account of the bending of a thin membrane on the package surface it is possible to measure the enclosed vacuum.

These potentialities of nondestructive measurements allow us to observe the fabrication of microstructures without unwanted influences.

## Location and magnitude of maximum strain in SMT solder joints

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Reliability prediction of SMT solder joints is based on low cycle fatigue due to cyclic thermo-mechanical stress. Thus the knowledge of the location and magnitude of the strain induced into a solder joint during thermal cycling is the fundamental requirement for any reliability estimation.

In this presentation the deformation behaviour of tin-based solder will be explained, the results of mathematical simulations will be presented and their impact on reliability predictions discussed.

## Local scanning probe methods

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During the last 18 years, the Scanning Tunneling Microscope (STM) has become an important instrument for the study of atoms and molecules of conducting samples.

Commercial Scanning Force Microscopes (SFM) have become popular to image the surfaces of samples from everyday life, in ambient conditions and with a higher resolution than with optical microscopes. There are large-scale applications in the quality control of hightech products such as IC's, CD's etc. Tribology and molecular recognition are other examples of applications for these instruments.

Only recently, the noncontact mode has opened a new field in which it is possible to observe true atomic resolution on insulators and to study magnetism and superconductivity.

Finally, there is a new development in which cantilever based nano-mechanics plays a major role. Impressive examples are: Bimetallic canti-levers for nanocalorimeters, cantilever sensors for the detection of stress in self assembled monolayers (SAM) and DNA hybridization, mechanical detection in ESR and NMR, and a mechanical Terrabit memory.

## The »size effect« on the fatigue – and fracture properties of thin metallic foils and wires

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Thin foils, sheets or wires of metallic, semiconducting or nonmetallic materials resemble essential parts of microelectronic and micro-electro-mechanical systems (MEMS). These parts are often subjected to mechanical and thermal fatigue causing premature failure. In numerous microdevices thin foils act as switches operating at frequencies from less than 1 Hz up to 1 MHz. Therefore the fatigue behavior of thin foils or wires attains considerable technical interest in order to assure a safe operation of MEMS. In the past mainly fatigue tests of given device have been performed. As the field of MEMS matured basic material properties of thin foils will be required.

Various investigations revealed that fatigue data of bulk materials cannot be adopted to almost two dimensional structures such as thin foils or films due to the »size effect«. From a summary of the most prominent fatigue testing and fatigue data of materials used in MEMS – recently presented at the »Fatigue 99« it may be concluded that the shapes of the specimens and/or their adjustments in the experimental set-up and the experimental procedure may be very different and sometimes rather complicated. Usually stress vs. fatigue life curves are measured in bending, also an equipment for tension-tension testing of thin Cu-films has been reported.

Almost no information on fatigue crack growth behavior of free-standing thin foils and wires is available.

Therefore, the objective of the present investigation was to develop a suitable testing method to investigate the crack growth behavior of thin metallic foils with thicknesses ranging from a few microns up to several micrometer and wires with diameters ranging between 10  $\mu\text{m}$  up to 150  $\mu\text{m}$ . The materials investigated were recrystallized rolled and electrodeposited Cu foils and Cu wires. The fatigue testing occurred using symmetrical push-pull loading at a fairly high test frequency of 20 kHz. Fatigue crack growth was investigated accompanied by the observation of dislocation structures due to plastic deformation in the vicinity of the crack.

To determine the static and fracture properties of the foils and wires a laser speckle extensometer to determine strain in combination with a microtensile machine was used. A strong size effect of the fracture strain was established being related to a characteristic fracture topography.

The results on the recrystallized rolled Cu foils indicate for thicknesses up to 100  $\mu\text{m}$  an unexpected crack growth behavior characterized by decreasing propagation rates with increasing crack length which may be explained by a transition of plane stress to plane strain with increasing foil thickness. This was confirmed by determining the ratio of the plastic zone related to grain size and thickness using special SEM-techniques. Intermediate crack growth arrests are due to a strong interaction of the crack with grain bound-

aries. The electrodeposited Cu foils showed due to the extreme fine grain size a similar crack growth behavior as bulk material.

The determined fatigue threshold stress intensity values of the foils were calculated under the assumption of LEFM. Generally the values are high which may be due to the ratio grain size, plastic zone and foil thickness. Results on fatigue life of Cu wires indicate a strong dependence on thickness. The fatigue and fracture mechanics data obtained from such thin structures allow to establish microfracture criteria.

## Determination of mechanical and tribological properties in the nm-range with the hysitron triboscope

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Mechanical properties of thin films and different phases in bulk samples often are difficult to determine. A new nano indentation technique is capable to determine hardness and modulus data from indentations with small indentation depth (0 – 500nm typical) and low applied forces (1µN – 30mN). With a scratch test friction forces and abrasive wear can be investigated.

The Hysitron Triboscope is capable to image the surface topography with the indenter when interfaced to a scanning force microscope (SFM). The indenter can be positioned with a high spatial resolution (<50nm) before the indentation test is performed. After the test the indented area can be imaged in order to observe surface cracking or thin film delamination.

The determination of mechanical properties of materials with high spatial resolution is especially interesting for MEMS devices and the field of micro-system technology.

As examples for applications of the new technique tests on different materials will be presented:

- Tribological properties of DLC-coating as protective coating for read and write heads of hard disks
- nano-indentations in a multiphase material
- nano-indentations on micro-structured material
- Determination of the spring constant of an AFM cantilever



## Nanofocus CT - A new dimension in radiosopic inspection on micro materials

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The state of the art in the field of nondestructive testing by X-ray technology related on spatial resolution is up to now given by so called open tube systems, i.e. systems in which the essential beam producing components like target or filament are rendered accessible by venting the tube.

The X-ray bremsstrahlung is created by impact of accelerated electrons focused on a target plane, which emit about 1 % of their kinetic energy (some 10 keV) during deceleration within the target. The emitted X-rays have a maximum energy limited by the maximum kinetic energy of the decelerated electrons. The rest of the electron energy is converted to heat which has to be dissipated via the target carrier.

The resolution  $d$  of a radiosopic systems is determined by detector resolution, magnification and focal spot size:

$$\delta = F \cdot (M-1)$$

with  $F$  = focal spot size and  $M$  = magnification.

Today the minimum focal spot size is within a range of 1 – 2  $\mu\text{m}$ . It is true that electrons can be focused to even much smaller spot sizes, however the mean free path length of electrons within condensed matter is about 1  $\mu\text{m}$  which means a real X-ray target spot of this size, even if the electron focus is much smaller (see fig. 1)

In order to achieve higher resolutions, new methods have to be developed. One of these methods is the cluster target which means that the electron focus will no longer be the limiting part, but the target plane is transferred or reduced to a target spot on a

target carrier with the size in the range significantly below 1  $\mu\text{m}$ .

For this a suitable heavy metal target (e.g. tungsten) of correspondingly small dimensions of some 100 nm will be embedded on a carrier produced from a material with a low nuclear charge number (e.g. beryllium).

The electron focus is then allowed to be larger than the

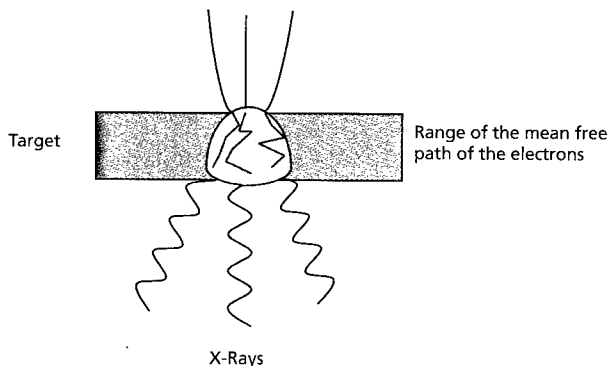


Figure 1: Schematic view of x-ray focus enlargement

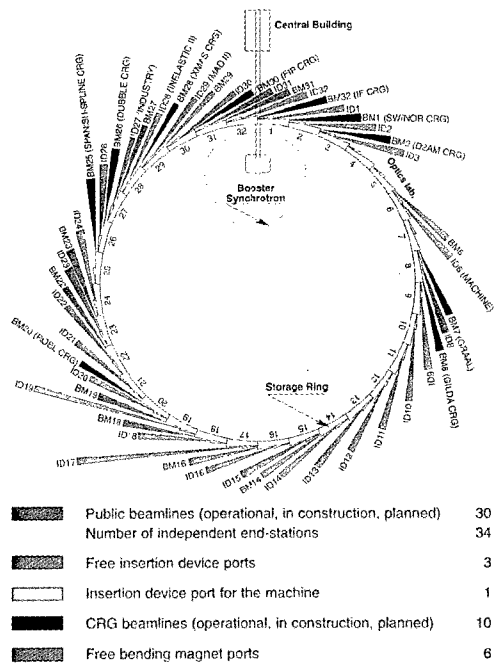


Fig.2: Beamline installation at the ESRF in Grenoble

target; due to the different materials of target and carrier, also different X-ray spectra are produced. The soft spectrum emitted by the carrier subsequently can be easily separated by suitable filters from the target spectrum and thus X-rays are resulting only from the small spot.

Another way for nanofocus X-ray sources is the use of synchrotron radiation. Synchrotron radiation facilities provide the most intense x-rays. High-energy electrons or positrons move within a storage ring and emit radiation whenever their path is curved; the curvature is induced either by a bending magnet or by an insertion device (see fig. 2).

The emission spectrum is well defined. Its calculation is based on a knowledge of the bending radius and the storage energy of the electrons or positrons. The

quality of the emitted radiation may be characterised by its brilliance.

$$\text{Brilliance} = N / \text{mrad}^2 \times \text{mm}^2 \times 0.1 \% \text{ bandwidth}$$

The brilliance describes the number of photons  $N$  emitted in one second from a source area of  $1 \text{ mm}^2$  into a cone defined by  $1 \text{ mrad}^2$  and normalised to a spectral bandwidth of  $0.1 \%$ .

In third-generation synchrotron sources so-called insertion devices are installed to improve the source characteristics. The electrons or positrons pass through a periodic magnetic field in order to increase the radiation power in a particular direction. Compared with a sealed tube, the gain exceeds six orders of magnitude.

A Brilliance of  $10^{19} \text{ c} / \text{mrad}^2 \times \text{mm}^2 \times 0.1 \% \text{ bandwidth}$ , an angular divergence of a few seconds of arc are possible, allowing to investigate small objects with high resolution.

This paper shows methods and results of present  $\mu$ -focus X-ray tomography down to a resolution of one micron, its transfer to the new fields of nano applications and the experimental setup of the nanofocus CT-machine and synchrotron beamline.

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## $\mu$ -XRF – A new powerful analytic tool for microstructures

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The necessity to conduct an elemental analysis or determine the coating thickness of very small areas arises at frequent intervals. Whenever this is the case, there are different conventional methods that can be used. Most of them use an electron beam for excitation, e.g. the Electron-Probe-Micro Analysis (EPMA). But EPMA has some disadvantages with respect to the preparation of electrical non-conductive samples as well as with respect to the sensitivity to small traces.

On account of these disadvantages, it is the X-ray excited fluorescence spectroscopy that renders the better analytical performance. However, in the case of this method, the concentration of excited radiation on a small spot proves a problem.

This problem can now be solved through the availability of new X-ray optics, with which it has become possible to concentrate the excited X-radiation on a small spot. The most common ones used in routine analysis are special capillary optics. With these optics, the diameter of the excited area can be reduced to the range of 30 – 100  $\mu\text{m}$ . Thus, the analytical performance of the EPMA can be improved significantly.

This improvement has the following implications:

- enhancement of sensitivity due to the non-existence of a background from bremsstrahlung
- the possibility to take measurements on air due to the fact that the excited radiation will not be absorbed in air
- no special sample preparation techniques for non-conductive materials are necessary
- analysis of lower layers of the sample is facilitated due to the excited radiation penetrating the samples better than electrons

Only smaller excited areas render it possible to analyze the distribution of elements by methods such as LineScan or Mapping.

In the paper, the qualities of capillary optics and their benefits for the performance of X-rays will be explained, and the characteristics of different types of capillary optics will be discussed.

Fields of application for this method of analysis for compositions and coating thicknesses of inhomogeneous samples are discussed.

## Multi chip power packaging - Reliability assessment

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Multi Chip Power Packaging describes a new packaging platform for mechanical relay replacement by semiconductor solutions, which has been developed in Motorola AISL-Europe. It offers additional control, diagnostic and communication functionality and is reliable at automotive test conditions. The fundamental idea involves the replacement of the existing copper heat sink in lead frame based power packages (i.e. HSOP, PSIP, or PFP) with a heat sink capable of supporting a circuit structure. The heat sink is constructed of a patterned Insulated Metal Substrate (IMS). This substrate material allows for one layer of electrical interconnection that is isolated from the heatsink, while providing adequate power dissipation capabilities. The use of flip chip devices was found to reduce  $R_{ds(on)}$  of Motorola Power MOSFETs and enhance the current carrying capabilities of the package.

This work assesses the mechanical reliability of the flip chip attachment on IMS. The associated solder interconnections are one of the key elements that define the device lifetime. Leading failure mechanisms and the significance of geometrical, material and process variables are described. Mechanical simulation, air-to-air thermal shock testing, cross sectional analysis and SEM/EDX analysis are used as main characterization tools.

## Photostructurable glasses with integrated wave guides

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The presentation will describe:

- a family of photostructurable glasses and glass ceramics with various thermal expansion coefficients
- structuring technologies based on a UV-lithographic process
- technologies for manufacturing optical wave guides in glass elements
- applications of components based on microstructured glass

The material properties of a photostructurable glass in the system  $\text{Li}_2\text{O} - \text{Al}_2\text{O}_3 - \text{SiO}_2$  will be presented.

The main process for structuring this glass is a UV-lithographic process. The structures which can be produced are two-dimensional and the depth is defined by the thickness of the wafer. Technological variations of the standard process will also be described in this paper.

The results of the technological variations are structures with a defined depth, structures with more than one definite depth or three dimensional structures free of undercut.

For applications in the range of microsensors and measurement, it is necessary to integrate an optical wave guide in a microstructured glass element. Various technologies are possible:

- thermal diffusion from a salt melt (e.g. silver ions)
- thermal diffusion in an electrical field
- structuring of a trench and filling with an other material
- implantation of ions by ion beams

Advantages and problems of the technologies will be presented and the use of the technologies for integration of an optical wave guide in a structured glass sensor will be described. New results of the technological investigation will be presented.

Some applications of microstructured glass elements especially in the range of microsensors and measurement will be shown.

## Experiments for the assessment of the biological effects of LCD materials on the environment

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Flat panel displays (FPD) are used nowadays in a vast number of electronic devices (consumer electronics, information technology, telecommunications, automotive, household appliances, toys etc.). For the year 2000 the production of FPD is expected to reach approximately 21 thousand millions US dollars. This corresponds to a quantity of more than thousand million pieces. With 85% share of the market Liquid Crystal Displays (LCD) are the dominating technology.

The use or life time of electronic products (e.g. mobile phones) in some cases is very short, therefore an increase of LCD as waste has to be expected for the future. To date, the ecological hazard resulting from these LCD materials is not known. According to the state-of-the-art waste LCD should be disposed of like hazardous waste. This kind of handling is based only on the assumption or estimation of adverse environmental effects of waste LCD. Resulting from this the possible environmental impacts of waste LCD are not investigated and clear concepts for environmental friendly handling of waste LCD are missing (Re-Use or Recycling).

In this paper measurements about the biological effect of LCD materials are presented. Waste LCD from industrial electronic recycling were extracted and eluted with water and organic solvents. The extracts were tested for inhibition of luminescence (DIN 38412 part 341) and growth (DIN 38412 part 37) with the luminescent bacterium *Vibrio fischeri*. A possible influence of heavy metals was assessed by the use of EDTA as complex forming agent.

Altogether the results show that the assessed extracts causes a considerable inhibition of luminescence bacteria i.e. a considerable biological impact. Further investigations will have to be done to identify the compounds of the waste LCD causing the inhibition. A significant contribution of heavy metals on the overall toxicity could be excluded.

## Determination of intrinsic stress during the growth of Fe(Zr) thin films

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We report about stress measurements during the growth of Fe(Zr) thin films using an optical deflection technique. The intrinsic stress built up for different film compositions is determined quantitatively. The Fe(Zr) thin films exhibit a phase transformation from an amorphous to a crystalline state if grown on early transition metal or rare earth substrates. The onset of the transformation is controlled by the film composition. The transformation can be followed quantitatively by determination of the structural and magnetic properties of the films. The transformation shows up in the development of the measured intrinsic stress: whereas the amorphous films exhibit very low intrinsic stress levels, stresses up to 1 GPa develop after the crystallization. The stress development in the crystalline state is associated with the microstructural evolution of the films.

## High Temperature Fiber Optic Thermal Device

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We have demonstrated a fiber optic thermal device capable of reaching temperatures as high as 1,800 °C. The device consists of a crystalline optical fiber onto one end of which is grown a doped tip of the same material. Laser power is launched into the other end, and is absorbed in the doped tip. When an appropriate dopant is used, the optical energy is converted efficiently into thermal energy. Because of the small dimensions of the tip, relatively little laser power is needed to heat it to a very high temperature. Because of the all crystalline construction, temperature approaching the melting point of the crystal can be achieved. This kind of device is potentially useful in microsoldering, microwelding, nano-scale surface modification, and micro-fabrication in general.

The prototype device was fabricated from a single crystal YAG fiber with a 5 at.% Nd:YAG tip. Both the fiber and the tip measured 300  $\mu\text{m}$  in diameter. A Laser Heated Pedestal Growth system was used to grow both. Starting with an initially tapered tip, it was possible to produce tips with varying size down to 30 nm through etching. Soldering of a gold wire with 25  $\mu\text{m}$  diameter to a 35  $\mu\text{m}$  wide gold trace on Kapton was accomplished with a tip measuring 20  $\mu\text{m}$  in diameter. A laser power of only 0.3 W was needed to form the joint. Details on the fabrication of the device and its implementation will be described.



## About the accuracy of the determination of mechanical properties of thin layers with the nanoindentation method

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*Physikalisch-Technische Bundesanstalt Braunschweig und Berlin, Germany*

The fast development of the nanotechnologies led to the fact that to the determination of mechanical properties of thin layers, e. g. hardness and elasticity, are put ever raising requirements. With the elaboration of the draft standards ISO/CD 14577-1 until -3 on the instrumented indentation test, which also describe the nanorange, essential foundations for the uniform application of the indentation method were laid. At this the nano range is defined for indentation depths  $h \geq 200$  nm, and if the rule of thumb is considered that the layer thickness  $d$  should be  $d \geq 10 h$ , the nanoindentation method can be applied for layer thicknesses  $d \geq 2000$  nm.

The paper reports on metrological investigations for the application of the nanoindentation method on thin layers.

In the first part, the accuracy of the universal hardness  $H_U$  is compared with that of the plastic indentation hardness  $H_{ind}$ . Generally,  $H_U$  can be determined more precisely than  $H_{ind}$ . The accuracy difference between the two hardnesses depends on the kind of tested material. But in the same time one has to consider that  $H_U$  characterizes the elastic-plastic and  $H_{ind}$  the plastic properties of the layer.

In the second part, the calibration of the indenters of nanoindentation measuring devices is examined. The indenter's geometrical deviations represent the most significant uncertainty source at nanoindentation measurements. The indenters on the one hand were calibrated with a modified scanning force microscope (SFM) and on the other with reference materials (e.g. fused silica). The modification of the SFM covered the incorporation of three laser interferometers into the SFM's coordinate axes  $x, y, z$ , thus realizing traceable measurements. The geometrical deviations of the indenter are summarized in an area function. The area functions received by the two methods are compared with each other. The obtained uncertainties of measurements due to the application of these area functions in nanoindentation measurements are given.

In the third part, the influences of the material combinations of layer and substrate on the result of the determination of the layer's hardness and elasticity are presented. At this an important influence quantity is the indentation velocity of the indenter. The variation of hardness and elasticity in dependence on the indentation velocity and the material combination of layer and substrate is discussed.

From the investigations conclusions are drawn for the further standardization of the nanoindentation method.

## Nano- and microscale materials in microreactors

Hessel, V.\*; Ehrfeld, W.\*; Löwe, H.\*; Richter, T.\*; Schiewe, J.\*; Zapf, R.\*;  
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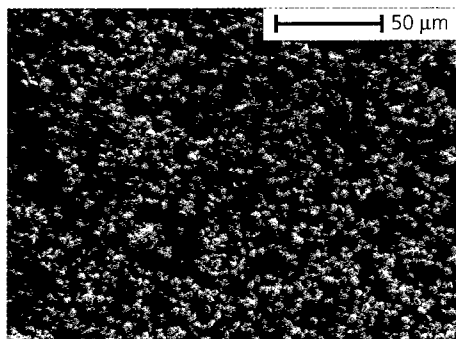
\*\* Institut DMX – LTP, EPFL: Ecole Polytechnique Fédérale de Lausanne, Lausanne, Suisse

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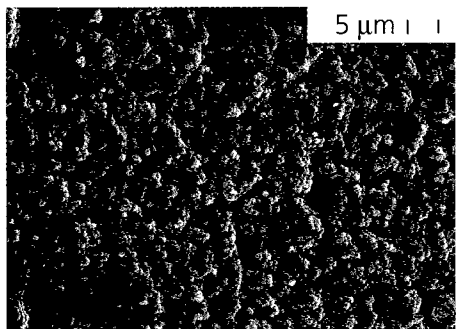
Miniaturized components and systems for performing unit operations and reactions in chemical technology, so-called microreactors, meanwhile are widely applied for process development and optimization in industry, universities, and institutes [1]. Recently, first applications of microreactors with respect to production, referring to fast polymerization processes [2] and metal organic reactions [3], have been described. Here, increased throughput is realized by parallel processing in microsystems.

Some of the current investigations are related to fundamental or applied research concerning the generation of materials with defined properties. This paper will discuss the generation of microscale materials by using microreactors as well as the modification

of construction materials for microreactors by advanced nanoscale deposition techniques.



Uniform copper oxalate particles generated in an interdigital micromixer



Porous α-alumina layer on a microstructured stainless steel substrate

The formation of microscale materials in microreactors presently is investigated in a number of activities focusing on various disperse systems, e.g. gas / liquid dispersions, emulsions, pastes, and suspensions. For this purpose, a large variety of micromixers meanwhile is available. Starting from a comprehensive overview on micromixing principles with respect to different flow configurations, two frequently applied approaches will be discussed in detail: The injection of many substreams of two fluids into a common mixing chamber (multilamination) and multiple splitting and recombination of a two component stream (split-recombine) [1]. As an example of this on-going development, micromixers suitable for volume flows up to 50 l / h currently being tested for their mixing and dispersing performance will be introduced.

Gas / liquid dispersions, emulsions, and

pastes with narrow bubble and droplet size distributions, respectively, are formed by means of an interdigital micromixer [4,5]. Target values like the average droplet size can be tuned by proper adjustment of influence quantities, e.g. of the volume flow of the fluids. In addition, microscale solid particles were generated by controlled precipitation of solutions in the same micromixer as an integral part of a segmented tubular flow system. First results of the synthesis of copper oxalate, formed by precipitation and agglomeration of nanoparticles [6], show a narrow particle size distribution of the microscale particles. By measuring the particle size distribution by a sedimentation method, a distribution comprising particles of a size of  $2.9 \pm 1 \mu\text{m}$  is obtained.

Nanoscale coatings, designated for use as catalyst carriers for gas phase reactions, were deposited by means of slurry and sol-gel techniques in microchannels made of various stainless steels. For alumina coatings, several types of modifications could be identified by means of infrared spectroscopy (FTIR), X-ray diffraction (XRD), and X-ray photoelectron spectroscopy (XPS). By variation of the process applied, nanoparticles ranging from 30 nm to 300 nm were achieved in 1  $\mu\text{m}$  thick layers. These nanoscale coatings were impregnated with several catalytic materials including the technically relevant combinations Pt / Zirconia, Ag / Alumina, and Cu / ZnO / Alumina.

At present, the applicability of these layers regarding reaction engineering is explored by means of CFD-simulations. These show that by using thicker catalyst / carrier layers in the order of about 100  $\mu\text{m}$  significant improvements in process performance, e.g. regarding the catalyst mass needed and system size reduction, are achievable for mass and heat transfer limited reactions.

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## New equipment for evaluating performance of microelements on MEMS

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Microelectromechanical systems (MEMS) consist of many microelements such as actuators, sensors and moving components. Evaluation of their performance including mechanical properties, generated force of actuators and sensitivity of sensors on MEMS devices are considered to be extremely important for design purposes. In this investigation, testing equipment for measuring not only the mechanical properties such as elastic modulus, yield stress and fracture toughness of microelements but also the performance of micro components such as the generated force of the actuators and mechanical response of sensors on MEMS has been developed. This testing machine consists of a magnetstrictive actuator which is able to produce a displacement of upto 10 microns with accuracy of 5 nm, a load cell with a load resolution of 10 micro-N and specimen stage which is able to move to adjust the testing point at a translation resolution of 0.1 microns. The actuator is connected to a metal shaft and a diamond tip of 5 microns in radius is attached to the end of the shaft. The load cell is installed between the diamond tip and the actuator. A MEMS device is set on the stage and an objective component is to be adjusted under the diamond tip from the actuator by moving the stage. The diamond tip is brought in contact with the objective component and mechanical properties are measured by the actuator and the load cell. If the component on the MEMS device is an actuator, the generated force is able to be measured by load cell during operation of the actuator on the MEMS. This equipment seems to be promising for evaluation of the overall performance in MEMS devices.

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## Automated optical measurements under vacuum

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Worldwide sensors get more and more applications also in safety systems. Thus the demands on the efficiency, the quality and the reliability of sensors increases. For inspection of diaphragm pressure sensors a flexible vacuum is useful. For this application the IZM have realized a automated evacuable measuring module for small to medium number of pieces.

The sensors or other components are automatically channelled into the measuring module, where they can be tested and examined. Furthermore the module enable to evacuate to the required vacuum. Simultaneously under vacuum, an electrical wiring-up of the sensors is possible, similar to a prober.

The next step is the measuring process. In addition to optical 3D-measurement techniques, a multispectral ellipsometer for the characterization of optical layers and a grey scale correlation for the determination of a surface shift can be used, e.g. for function tests.

The whole process, from the input of measuring object over to the measuring process up to the output, occurs automatically, likewise a corresponding »superior/inferior«, quality decision can be obtained.

## Analytical simulation tools to determine the behavior of bulk and layered substrates

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Analytical computer simulation tools have been developed to analyze both isotropic and thin film layered materials under indentation and scratch loading conditions. The models developed predict the mechanical behavior of these material systems to assist engineers and scientists determine the stresses and displacements resulting from concentrated loads.

The basis for our simulation models are founded in the original work performed by Hertz to analytically determine the hemispherical contact pressure resulting from a circular contact. [1] Subsequently sliding contact problems were refined by Hamilton and others. [2,3] The work presented has been extended to predict the stress distribution in bulk and layered materials under both axial and transverse loads from spherical, conical, and Berkovich tip geometries. Utilizing Fabrikant's [4] mathematical simplifications, we have developed a method to accurately predict the load versus displacement behavior of materials under axial and transverse loading conditions. Within the elastic limit, the simulation models described herein accurately predict the bulk indent and scratch properties of materials composed of thin layered materials up to approximately ten layers with a variety of material properties. We have compared our results to two research groups and find agreement within 4% for indentation test models. Additionally, we will present a comparison of scratch test simulation results to tests performed with our instruments on both isotropic and layered substrates that represent industrial applications.

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## The micro stereoimage strain analysis by the microscope Zeiss Neophot 21 with Praktica Scan 2000

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Optical methods of strain measurements represent one of the oldest and the most widespread area of experimental mechanics. The optical methods of strain measurements are generally interferometry, stereo image analysis and digital image analysis. The above-mentioned optical methods have particular importance for deformation analysis in microstructures and microobjects, where they are the only possible experimental tools.

The basis of the stereo image analysis of deformation measurements is the taking a pair of stereoscopic photographs of objects, using the time base principle, i.e. photographs of objects are taken before and after a deformation and under the entirely same conditions. The further step in stereoimage technique of the strain analysis is the comparison of the pair of images. The glass photoplates and the optical stereocomparator were used recently for this purpose.

The application of digital analysis in this area means to work with the digitalized images of microstructure. The comparison of the pair of digitalized images must operate with them on the one pixel level. The requirement of a high accuracy comparison of the digital images needs to have ones with a great number of pixels in the X and Y directions too. It requires to use the recording equipment with the high resolution and currently hardware of the computer must be able to process large images.

The images taken by the optical microscope Zeiss Neophot 21, which is used, can fulfill the foregoing accuracy condition, if the digital camera Praktica Scan 2000 is used as a recording medium. Its maximum resolution is given by the image size of  $3648 \times 4625$  pixels. The comparison accuracy of images on the monitor, then depends on a good focusing of the camera and on a good, enough sharp, marking of the specimen surface. This problem we resolved by setting of the additional CCD camera with intermediate lens and by specially made reduction on the viewfinder of the photoscanner. By means of this camera and using the VideoTip programme, we can focus the image in a large zoom directly on the monitor of the computer. The communication of the photoscanner with the computer is realized through the SCSI interface, which makes possible a fast reading of the images. The plug-in, named SilverFast, was developed by the firm LaserSoft, for importation of the images through the TWAIN interface into the Photoshop software.

The deformation analysis of the pair of digital images is then carried out by using the stereoimage software Comparat. This software enables to read the images before and after the deformation into the programme and allocate appropriate pixels from the first and the second images. The output from the Comparat is the text file with X and Y coordinates of associated pixels. The strains are then calculated from deformation gradients, which are identical with the coefficients of affine transformation written for

coordinates of both images. Examples of the analysis of residual strains – by using of small size ring-cutting method – and localized deformations – grains rotation, are presented.



## Lead free alternatives: An analysis of performance characteristics

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Due to the toxicity of lead, there is a tremendous amount of effort on eliminating lead from the solders used in electronic industry. The move toward lead-free solder alternatives in North America and Europe accelerated significantly since Japanese industry announced their aggressive lead-free roadmap. For instance, Toshiba, Matsushita, and Hitachi have announced plans of elimination of all lead interconnects in one or all products by 2001, 2004, and 2004, respectively. However, the preferred solution for lead-free alternatives varies from region to region, and there is a number of alloys considered promising. The most favorable Pb-free solder systems identified by the industry comprise primarily alloys of Sn with Ag, Bi, Cu, Sb, or Zn, such as 99.3Sn0.7Cu, 96.5Sn3.5Ag, 95.5Sn3.8Ag0.7Cu, 93.6Sn4.7Ag1.7Cu, 96.2Sn2.5Ag0.8Cu0.5Sb, 91.7Sn3.5Ag4.8Bi, 90.5Sn7.5Bi2Ag, 89Sn8Zn3Bi, and 58Bi42Sn. Unfortunately, most of the promising alloys were evaluated under a single flux system. The compatibility between flux and alloy often dictates the performance of reflow soldering, such as solder balling, wetting, processing window, and stability. Since the flux chemistry varies from supplier to supplier, and since the use of more than one suppliers is considered crucial for assuring a steady process, an alloy being compatible with a wider range of flux systems obviously will have a greater prospect to be accepted by SMT industry. In this study, a group of most promising Pb-free alloys reported are tested against a broad range of commonly used flux chemistries, such as water wash or no-clean, halide-containing or halide-free, nitrogen reflow systems or air reflow systems, in the form of solder paste. The reflow soldering performance of those paste is evaluated and ranked in order to assess the prospect of those alloys being widely used for reflow soldering applications by the industry.

## The fictitious indentation depth-load trajectory as a new approach for determining thin film mechanical properties without substrate effects

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The indentation test has been developed to a popular method for investigating mechanical properties of thin films. However, there exist only some empirical or semi-analytical methods for determining the hardness and the Young's modulus of a film from pyramidal indentation of the film on a substrate. In the present paper, a new and more general concept is proposed using a depth-load trajectory which is related to a fictitious *bulk film* material (see Fig. 1). This *bulk film* material is supposed to possess the same mechanical properties as the real film. It is assumed that the film and the

substrate exhibit elastic-plastic material properties with nonlinear isotropic and kinematic hardening. The determination of the depth-load trajectory of the bulk film is a so-called inverse problem. For solving this inverse problem IP2, use is made of the method of neural networks. Having established the bulk film depth-load trajectory, the set of material parameters entering in the constitutive laws may be determined by solving the inverse problem IP1 (see Fig. 1) using methods, developed previously [1,2].

The inverse Problem IP2 is

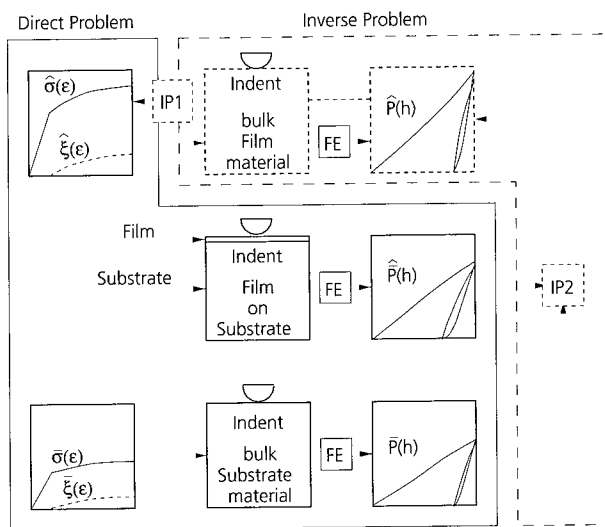


Figure 1: Concept for solving the inverse problem.

solved using two neural networks, denoted as LoadNet and HystNet. The input and output quantities are required and expressed in dimensionless form, respectively. Thus, arbitrary indenter radii can be used. LoadNet determines the loading response  $\hat{P}_l(h)$ , the maximum load  $\hat{P}_t$  at  $h = h_t$  and the unloading stiffness  $\hat{S} := d\hat{P}_u/dh|_{h_t}$  from the corresponding depth-load responses for the pure substrate and film on substrate. HystNet determines the values of opening and width of the fictitious film hysteresis at three equidistant positions between  $\hat{h}_t < h < h_t$  and  $0 < P < \hat{P}_t$ , respectively. The quality of the trained networks has been validated using additional test examples, which were not presented to the networks during training.

Having established the load response and hysteresis loop geometries of the fictitious bulk film material, the whole inverse problem is accomplished by applying the existing solution method IP1 for the homogeneous problem to determine the material parameters of the real film.

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## Patterning of thin plasma polymerized fluorocarbon films

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Direct patterning of fluorocarbon (FC) films is very difficult due to the low free surface energy of the FC-polymers. The conventional lithography process is often unsuitable. The spin coated resist cannot adhere on the FC-surface and it is impossible to realize the lithography process. The aim of this study was to find out another possibilities to pattern thin plasma polymerized FC-films. For this reason, various materials (photoresist and metals) were investigated as masks. Different spin-coating parameters were examined to determine the optimum values of the lithography process when FC-films were pattern trough resist masks. The influence of the metals on the surface properties of the FC-films (e.g. surface free energy) were examined when metals were used as masks. A lift-off process were also investigated and used as technology for patterning of the FC-films. The lift-off process have shown the best results (high resolution, unchanged surface properties of the FC-films, low surface free energy after the patterning).

## The impact of research and market on the economic success of small and medium sized enterprises (SMEs)

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In today's business environment entrepreneurs recognise the crucial importance of R&D-activities and technology transfer to the competitiveness of their companies. This situation is profoundly experienced among SMEs which depend upon rapidly changing technologies for their source of competitive advantage.

Research partnerships between academic institutions and industry play a key role in the strategic approach to innovation. Public funding has facilitated the creation of regional, national and international partnerships enabling business competitiveness and regional economic development.

Product differentiation and diversification are success factors for SMEs. Profound market knowledge is of importance for strategies that are focussed on customer orientation. Limited capacities and lack of funds are the main obstacles in implementing marketing instruments.

Research activities and market analysis are incorporated in a complex managerial process. SMEs are challenged to manage technological change, to understand new markets, to adapt to emerging technological infrastructures and – last but not least – to gain profit.

This presentation will concentrate on success factors and financial strategies in the microtechnology field.

## Non-equilibrium processing of thin Si films via pulsed laser irradiation

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Thin Si films on a variety of substrates are becoming increasingly recognized as a technology-enabling material for a variety of large-area electronic applications. The specific microstructure of the utilized films can range from amorphous and microcrystalline for photovoltaic devices to large-grained polycrystal and single-crystal for high-performance thin-film transistor devices.

In this paper we show that non-equilibrium processing of Si films via pulsed laser irradiation represents an extremely flexible and potent means of manipulating and controlling the microstructure of the films. In particular, it is noted that the method can be used not only to crystallize amorphous films and amorphize crystalline films, but also is capable of changing the microstructure of the crystalline Si films (e.g., microcrystal to single crystal or single crystal to microcrystal, etc.).

In order to correctly interpret and optimally utilize all these phenomena and capabilities, we discuss how it is necessary to first recognize that all the transformations that are induced by short-pulse-duration laser irradiation are melt-mediated first-order phase transitions, and additionally that they occur under extreme conditions that are substantially removed from equilibrium. As such, a proper analysis of the process requires – in addition to the thermal considerations during heating, melting, and solidification – the competitive kinetic considerations involving the interfacial temperature-dependent motion of the phase boundary and the liquid temperature-dependent rate of solid nucleation within the deeply supercooled melt.

We will elaborate on, as an example, a particular form of pulsed-laser-based crystallization method referred as sequential lateral solidification (SLS) that permits realization of low-defect-density crystalline Si films with controlled microstructures on glass or plastic substrates; such materials are desired in various microelectronic and large-area-electronic applications.

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## Dicing of silicon wafers

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### 1 Introduction

Dicing is the final process in the semiconductor device manufacturing that separates the silicon wafer with integrated circuits into individual chips. This means that dicing is the process conducted to highly value added materials being requested to be extremely reliable. On the other hand, the strong demand to increase the productivity of semiconductor device manufacturing requires the dicing process be highly productive. To meet the demands to increase the productivity as a whole, feed rate of the silicon wafer should be increased. However, this leads to some serious troubles such as chipping of the wafer on the kerf, excessive blade wear and meandering of the diamond blade. This paper deals with the above mentioned troubles in the dicing process aiming at making clear the influence of setup parameters on those troubles and consequently to make the practical suggestion for improving the productivity of the dicing process.

### 2 Chipping of Silicon Wafer

When the wafer is diced, micro chipping appears on the kerf. The chips become scrap if the chipping interferes the patterns on the wafer. Therefore, chipping should be suppressed as much as possible. In order to investigate the influence of the dicing conditions on the chipping of silicon wafer and eventually to prevent the chipping, a series of experiments was conducted. The maximum kerf width increases with decrease in the blade speed and slightly increases with increase in the feed rate. In order to make more clear the influence of the dicing parameters on the chipping of wafer, a statistical analysis based on the design of experiment was carried out. It was confirmed that the influence of the set-up parameters such as the blade speed, the feed rate and the depth of cut was significant in comparison with the specification of the blades.

### 3 Meandering of the Diamond Blade

The kerf generated on the silicon wafer should be straight and not interfere the patterns. However, this requirement is not necessarily always satisfied and the kerf waves due to the meandering of the blade. When the meandering phenomenon is significant, the diamond blade is plastically deformed and eventually broken.

The blade speed is the most decisive factor for this phenomenon; when the blade speed exceeds a certain critical limit, meandering starts to occur. In addition, the protrusion of the blade from the flange also has a decisive influence. From a series of experiments, it was envisaged that the meandering was a kind of instability phenomenon.

#### **4 Wear of Diamond Blade**

Radial wear of the diamond blade is crucial in dicing because the protrusion of the blade from the flange can not be large enough to keep the blade stiffness. The amount of the protrusion is around 1 mm, and when the radial wear reaches this amount, the blade should be replaced with a new one. Thus, the excessive blade wear results in the increase of non-machining time for blade replacing.

The wear increases almost in proportion to the dicing length, while it is accelerated with increase in the feed rate and decrease in the blade speed. The influence of the blade specification on the wear is not so significant as the dicing setup parameters.

#### **5 Dicing Forces**

Measurement of the dicing forces must provide us some important information to understand the process. A KISLER dynamometer was installed to the dicing machine to make the measurement of three force components possible.

The influence of dicing parameters on the specific tangential dicing force  $F_t$  can be roughly represented by the equivalent chip thickness ( $v_w a_p / v_s$ ) as

$$\frac{F_t}{b} = \alpha \left( \frac{v_w}{v_s} a_p \right)^\epsilon, \quad (1)$$

where  $b$  is the blade width,  $\alpha$  is the constant,  $v_w$  is the feed rate,  $v_s$  is the blade speed,  $a_p$  is the depth of cut and  $\epsilon$  is exponent.

#### **6 Conclusions**

Some troubles in dicing of silicon wafer, such as chipping of the wafer, meandering of the blade and its wear were experimentally investigated. Increase of the blade speed is effective to suppress the wafer chipping and the blade wear. However, the meandering of the blade that appears to be a kind of instability is apt to occur when the blade speed increases.



## Production of high precision microparts for machines using metallic glass

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Since a series of discoveries of new glassy alloys with a large supercooled liquid region before crystallization in Mg-, Ln- and Zr-based alloy systems for several years between 1988 and 1990, the glassy alloys have attracted intense attention because of the formation of bulk glassy alloys with superplasticity in the supercooled liquid. It has subsequently been reported that bulk glassy alloys with a large supercooled liquid region are also formed in Zr-Be-, Fe-, Pd-Cu- and Ti-based systems. More recently, we have systematically examined viscous flow behavior of the Ln-, Zr-, Pd-Cu- and Fe-based glassy alloys as a function of temperature and strain rate. As a result, it has been reported that the flow stress ( $\sigma$ ) of the supercooled liquid increases almost linearly with increasing strain rate ( $\dot{\epsilon}$ ) and the strain-rate sensitivity exponent ( $m$ -value) defined by the slope of the linear relation is approximately 1.0. The viscosity ( $\eta$ ) of the supercooled liquid evaluated by the relation  $\eta = \sigma_{13} / \dot{\epsilon}$  remains constant in a wide strain rate range below about  $0.1 \text{ s}^{-1}$  and decreases almost linearly with further increasing strain rate, indicating the transition from Newtonian to non-Newtonian flow. It is noticed that Newtonian flow is maintained even at the high strain rates up to  $0.1 \text{ s}^{-1}$ . The Newtonian flow is obtained only in the supercooled liquid region and changes into the non-Newtonian flow with decreasing temperature. That is, no Newtonian flow is obtained in the glassy solid. The maximum elongation is obtained in the Newtonian flow state at the high strain rate just below the transition from Newtonian to non-Newtonian flow and the largest elongation is about 1800 % for the La-based alloy, larger than 1300 % for the Pd-Cu-based alloy, 280 % for the Zr-based alloy and 240 % for the Fe-based alloy. Even after the deformation up to the maximum elongation, all the alloys keep good bending ductility. One can notice that the supercooled liquid in all alloy systems exhibits large elongation above 200 % at high strain rates above  $0.01 \text{ s}^{-1}$ , indicating the satisfaction of the criterion for the high-strain rate superplasticity. By utilizing the high-strain rate superplasticity which cannot be obtained for crystalline superplastic alloys, the bulk glassy alloys can be deformed into micro-materials such as optical mirror, gear, connector, spring, fiber and thin sheet etc. The resulting glassy metallic micro-materials have been expected to be used in some application fields.

## High-strain rate superplasticity due to Newtonian flow of supercooled liquid in bulk glassy alloys

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Since a series of discoveries of new glassy alloys with a large supercooled liquid region before crystallization in Mg-, Ln- and Zr-based alloy systems for several years between 1988 and 1990, the glassy alloys have attracted intense attention because of the formation of bulk glassy alloys with superplasticity in the supercooled liquid. It has subsequently been reported that bulk glassy alloys with a large supercooled liquid region are also formed in Zr-Be-, Fe-, Pd-Cu- and Ti-based systems. More recently, we have systematically examined viscous flow behavior of the Ln-, Zr-, Pd-U- and Fe-based glassy alloys as a function of temperature and strain rate. As a result, it has been reported that the flow stress ( $\sigma$ ) of the supercooled liquid increases almost linearly with increasing strain rate ( $\dot{\epsilon}$ ) and the strain-rate sensitivity exponent ( $m$ -value) defined by the slope of the linear relation is approximately 1.0. The viscosity ( $\eta$ ) of the supercooled liquid evaluated by the relation  $\eta = \sigma_{13} / \dot{\epsilon}$  remains constant in a wide strain rate range below about  $0.1 \text{ s}^{-1}$  and decreases almost linearly with further increasing strain rate, indicating the transition from Newtonian to non-Newtonian flow. It is noticed that the Newtonian flow is maintained even at the high strain rates up to  $0.1 \text{ s}^{-1}$ . The Newtonian flow is obtained only in the supercooled liquid region and changes into the non-Newtonian flow with decreasing temperature. That is, no Newtonian flow is obtained in the glassy solid. The maximum elongation is obtained in the Newtonian flow state at the high strain rate just below the transition from Newtonian to non-Newtonian flow and the largest elongation is about 1800 % for the La-based alloy, larger than 1300 % for the Pd-Cu-based alloy, 280 % for the Zr-based alloy and 240 % for the Fe-based alloy. Even after the deformation up to the maximum elongation, all the alloys keep good bending ductility. One can notice that the supercooled liquid in all alloy systems exhibits large elongation above 200 % at high strain rates above  $0.01 \text{ s}^{-1}$ , indicating the satisfaction of the criterion for the high-strain rate superplasticity. By utilizing the high-strain rate superplasticity which cannot be obtained for crystalline superplastic alloy, the bulk glassy alloys can be deformed into micro-materials with various outer shapes. The resulting glassy metallic micro-materials have been used in some applications fields.

## Molecular modeling and discrete element modeling applied to the microelectronics packaging industry

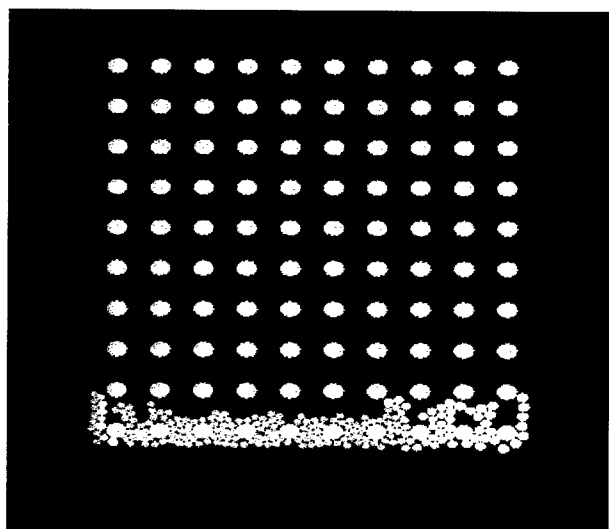
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At Allied Signal we have been studying the use simulation techniques in order to understand the underlying physical and physical chemical principals correlated to material properties such as wetting, adhesion, flow, particle compaction and interfacial reliability. In order to do this we have employed two basic modeling techniques: molecular modeling and discrete element modeling. Whereas molecular modeling is a well-established simulation tool found primarily in the pharmaceutical industry, discrete element modeling is a newly emerging simulation technique and represents an opportunity to link the molecular scale to meso-scale engineering processes. The first step in our work at Allied Signal has been to link the molecular structure to the physical chemistry on a molecular scale. These analyses are directly applicable to the formulation chemist interested in understanding or refining his material, as basic structural and compositional changes may be made at the molecular level and correlated to a property or process. Such calculations have been especially helpful in understanding specific interfacial characteristics from the molecular level in which local energy effects create properties different than the bulk. These characteristics are especially important in understanding the effects of contamination and moisture on wetting and adhesion, which will ultimately affect reliability. We have also found that the energy profiles obtained in these studies can then be applied to the discrete element model in order to

attain a more physically correct picture of the meso-scale dynamics in the working material system, especially as applied to an engineering process. Specifically, we have used discrete element modeling to describe underfill flow and silver filler compaction. An example of an underfill flow simulation in a 10X10 array of solder bumps is shown in the following figure in which the particle adhesion, as determined from the molecular scale, has become important enough to



obstruct the flow of particles between the bumps. These studies have given us a better understanding on the impact of the binder (carrier) to filler relationship. We believe that these types of approaches will be more important as feature sizes shrink within the board and packaging industries and interfacial characteristics rather than bulk characteristics begin to play a larger role in failure. This paper will give an overview of the lessons learned in this ongoing work.

## Properties of diamond for sensor applications

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The extreme physical, optical and electrical properties of diamond have been recognised for some years, but natural forms of this material are unsuitable in terms of size and cost for sensor applications. Moreover, natural diamond is a highly variable substance with few stones exhibiting the extreme optical and electrical properties required for high performance sensor operation. However, the emergence of large area thin film diamond, grown by CVD techniques, in the late 1980's opened up the prospect of commercially interesting sensor fabrication from this material. Despite years of intensive research thin film diamond grown on non-diamond substrates remains a polycrystalline material which plays host to a wide range of defects. The realisation that the growth of single crystal diamond by CVD techniques (on non-diamond substrates) may not be possible, allied with the difficulties encountered when attempting to electrical »dope« this material, led to a significant reduction in expectations for the practical use of thin film diamond for commercial sensor applications in the mid-1990s. This paper will describe recent advances in the technology associated with doping diamond and show how defects that form when the diamond is grown can be passivated through the use of high temperature gaseous treatments. These improvements are such that at the beginning of the 21st century we can again expect diamond sensor technology to have a real commercial impact. Indeed, UV detectors produced to the authors design are being produced under licence by industry, one of the first examples of the commercial acceptance of this material.

The wide band gap of diamond (5.5eV), allied to its high resistivity imply that it is ideally suited for the fabrication of deep UV (<225nm) sensors that would be »blind« to visible radiation. Despite this promise, early attempts to realise this type of detector were dissappointing. We have recently demonstrated that the use of defect passivation treatments enables high performance photoconductive detectors to be constructed; high levels of gain ( $10^6$ ) and speed (1GHz) can be achieved allied to low dark current (0.1nA) and visible blindness ( $10^6$  discimintaion). The realisation of these structures will be described. The formation of more complex electronic devices requires that the diamond is doped; recent work on the hydrogenation of CVD diamond films has shown that p-type material can be generated this way, producing carriers with a low activation energy (meV, compared to 0.37eV for boron) and a high mobility ( $\sim 70 \text{ cm}^2/\text{vs}$  for  $5 \times 10^{17}$  carriers). An activated trapping process occurs, meaning that at low temperatures there are actually more carriers, prior to freeze out <15K. Using this approach we have fabricated optically activated field effect transistors, whose properties will be described.

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## Thermo-mechanical testing of polymeric materials under rates of deformation between $10^{-2}$ m/s – $10^{+2}$ m/s

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In modern applications polymeric materials are often subjected to extremely mechanical stress in short time scales. The thermo-mechanical behaviour under these conditions is mostly unknown and concrete material principles do not exist. The properties of polymeric materials at high deformation rates are only scarcely available but are important for the simulation and modelling of stability of structures (Finite Element Method) and for the creation of new special materials (Composite Materials) with innovative characters and for high tech applications. These properties are also important for the science around the description of the thermo-mechanical behaviour of polymeric materials under high deformation rates. The mechanical testing of polymeric materials under these conditions requires a sophisticated measurement technique. Strain rates from 1 m/s up to 100 m/s make the measuring of relevant material-parameters difficult, which are needed to describe the mechanical behaviour. A special problem arises when measuring the local deformation rate and the energy flow through the material. A solution will be the use of different high speed cameras, operating in an analogous or digital modus.

The preference of the digital High Speed Video Technique takes into account the immediate availability of data necessary for the image processing. Combined with a special software to visualise flow phenomena in a vector representation, the combination allows to get material values, such as elongation, Poisson ratio and Young's modulus out of a vector field calculation. In this way, it is important to observe special points which must not necessarily exist in a regular pattern. The comparison of the patterns of two successive frames, allows the determination of the flow field of moving points. Thus it is possible to derive the superficial flow field of the test sample. Regarding time, the limiting factor is the used High Speed Video Technique, which provides only 4.000 frames/s, a restricting resolution with respect to space and time. The analogous measurement technique »Hycam« has not such limiting factor. This technique works with a rate up to 40.000 frames/s. There exists only the problem to transform all the interesting frames into a digital structure for the image processing. A third measurement technique is used to determine the thermo-mechanical behaviour under same conditions. The »Thermo-Camera« works with 3.500 frames/s in a temperature scale from  $-10^{\circ}\text{C}$  up to  $+2.000^{\circ}\text{C}$ . So it is possible to observe the energy flow through the polymeric material during a deformation experiment. The measurement of the energy distribution inside the breaking zone (Gradient of temperature) or the conversion of mechanical energy into thermal energy and the release of chemical energy during a breaking process contain information regarding the chemical boundaries, the throw off of the polymeric chains and different other information related to the material behaviour

depending on thermo-mechanical aspects. In all cases, the main interest according to the correlation between the in coupled force and the thermo-mechanical behaviour of the polymeric materials is, to find a relation which describes the behaviour of polymeric material under high deformation rates and elongation.

## Determination of critical interfacial energy release rate in hard coatings on ductile substrate

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Hard coatings such as diamond, DLC and TiN have been used for wear-resistant, lubricant and protective coatings, etc. However, these severe stresses exerted by external environments during service cause the film debonding and then deteriorate its workability. Thus, the study for improving the adhesion of film has become important, which requires the exact measurement of interface adhesion. Recently, instead of strength-based test methods often used in previous age, the study to obtain the interface fracture toughness or critical energy release rate is more active. In this study, two analysis models will be proposed for the adhesion measurement, models on cantilever beam specimen and indentation cracking method. The former specimen is divided into loading part with only single beam and crack propagation part with 4 layers beam such as substrate, film, adhesive and crack-driving attachment. In the specimen, the energy release rate with crack propagation is calculated using beam bending theory and compliance method. At this time, merits of the method are that the energy release rate can be calculated without knowledge of film elastic modulus and thickness and the propagated crack length can be predicted from load-displacement without direct measurement. Compared to former, the analysis of so-called indentation cracking test is more complicated due to various types of indenter and complex indentation stress field. The proposed model is about multi loading and multi measurement of crack length instead of single loading. The film is debonded from substrate loaded under various loads by Brale C indenter and then the crack length is measured by image analyzer system. Almost linear relation between indentation load and crack length, which is perhaps thought to be closely interrelated to interface adhesion, is obtained through the experiment. In the model, critical interfacial energy release rate is quantified from the experimentally-determined slope of load versus crack length, using the accumulated knowledge of indentation stress field involving plastic deformation of the substrate.



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## Technologies and materials for microsystems

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The VDI/VDE-Technologiezentrum Informationstechnik GmbH (VDI/VDE-IT) is an independent organisation founded by two of Europe's major scientific and technical organisations VDI and VDE.

Its main objective is to support industry and science in exploiting and transforming R&D results into innovative products, industrial processes and services, with a special emphasis on selected areas of information technology. Its mission focuses not only on technological developments but also on related economic and social implications.

VDI/VDE-IT operates at regional, national and European levels. It participates in the design and is responsible for the implementation and management of innovation support programmes run by the Federal Ministry of Education and Research (BMBF) and the Federal Ministry of Economics.

VDI/VDE-IT has been in charge of the support programme Microsystem Technologies on behalf of the German Federal Ministry of Education and Research since 1990. This programme will be continued beyond 1999. The extensive potential of microsystem technologies must be further opened to the area of new product developments. Support will be focused on economically relevant fundamental application areas. In Germany high potential for application can be found in the fields of mechanical engineering, construction of new installations, environmental technology, household technology, nutritional technology, automotive technology and medical technology. Technological development has reached a point where even standardised components can be produced.

The development of new microsystems needs the optimisation of technologies and of special micromaterials too. In order to realise new functions in the field of microsystem technologies there are special requirements e.g. for polymer with new active functions, new adhesives for packaging and interconnection technology, electro- and magnetorheological materials for actuators and shape memory alloys.

## B-doping and piezoresistivity of CVD diamond

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A market study of the total available market for high temperature electronics estimates that by the year 2005 roughly 4.7% of high temperature applications will require devices operating at temperatures in excess of 300 °C. According to this study the most important markets for high temperature electronics are well logging, aerospace and automotive applications. Wide bandgap semiconductors like diamond, SiC and III-nitrides are attractive materials for temperatures beyond 300 °C where conventional devices cannot operate.

The figures of merit for diamond are superior to those of all other semiconductors and diamond devices could theoretically demonstrate outstanding performance. Apart from diamond's ability to operate as a semiconductor at high temperatures diamond is chemically inert against environmental influences, mechanically hard, stiff and radiation hard. Such outstanding physical properties lead workers to consider the application of natural diamond stones as potential radiation detectors over 70 years ago. So far the lack of large area single crystalline substrates and the lack of a significant, controllable n-type conductivity limits the number of possible devices. However, with the advent of improved chemical vapour deposition of large area diamond films new applications become possible. Currently, thermistors and pressure sensors are being tested at the engineering prototype level. It is expected that diamond sensors for high temperature applications will penetrate into the market soon.

This presentation concentrates on piezoresistive sensors. The relationship between doping and piezoresistivity for diamond will be highlighted and its temperature dependence analyzed.

## Thermal Management with Novel Materials

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The efficiency and behaviour of the thermal management solution dictates the reliability of modern electronic systems. It is no longer sufficient to provide a mere temperature reduction but one must look to control the whole system and in particular the thermal expansion behaviour. The drive to provide good engineering solutions to meet the ever increasing demands for improved reliability has led to the development of new materials, including metal matrix ceramic composites and CVD diamond. This paper will show how these materials have been exploited to deliver reliability and performance benefits.

## Materials characterization on a nanometer scale at BESSY

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Insight into the microscopic world exploiting the unique properties of synchrotron radiation has a long tradition at BESSY. X-ray microscopy for investigating biological samples as well as combinations of X-ray-spectroscopy techniques with lateral resolution for the analysis of solids, composite materials, thin films etc. are currently available at BESSY for materials research on a sub-micrometer scale. Some of these techniques are well established at BESSY and are frequently used for basic research and industrial applications.

Putting the third generation synchrotron radiation source BESSY II into operation, the window is opened for materials characterization with a spatial resolution on the nanometer scale. To exploit the high brightness of the synchrotron light at BESSY as well as its polarization properties, the experimental techniques already available need to be improved further. In addition, new techniques like nano X-ray fluorescence or nano-EXAFS will be developed. In the near future, BESSY will offer materials characterization capabilities combining a large variety of spectroscopic techniques with a lateral resolution on the nanometer scale.

An overview will be given of the microscopy capability already available at BESSY. The current activities to exploit the high brilliance, the tuneability and the polarization properties of the synchrotron light will be presented, and plans for future work pushing the spatial resolution further down will be discussed.

## Nanocrystalline hydride electrode materials

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Metal hydrides such as  $AB_n$  (A - rare earth or transition metal; B - transition metal;  $n=1, 2$  or  $5$ ) and related substituted alloys have been extensively studied in recent years. These intermetallic compounds belong to the most promising electrode materials for application in rechargeable Ni/MH<sub>x</sub> batteries because of the rapid and reversible storage of large quantities of hydrogen [1]. Conventionally the metal hydride materials have been prepared by arc melting and annealing. New processing method such as mechanical alloying (MA), where the kinetic energy of colliding balls is used to mill, deform and cold-weld alloy powders is new way for producing hydrogen absorbing alloys.

Among the hydrogen forming compounds,  $AB_5$ -based intermetallics have recently proven to be very attractive as negative electrode materials. The merit of these compounds is that they exhibit low hysteresis, are tolerant to gaseous impurities and are easily hydrogenated in the initial cycle after manufacture. The properties of hydrogen host  $AB_5$  materials can be also modified substantially by alloying, to obtain the desired storage characteristics, e.g. proper capacity at a favourable hydrogen pressure. For example, it was found that the respective replacement of Ni in  $LaNi_5$  by small amounts of Al resulted in a prominent increase in the cycle life time without causing much decrease in capacity [2]. The use of an unrefined rare earth mixture (Mm - mischmetal) is very effective in lowering the cost of  $MmNi_5$ -based alloys.

In this work, the formation of nanocrystalline  $AB_5$ -type phases by mechanical alloying are reported. The structural and electrochemical properties of a range of alloys, including  $LaNi_5$ ,  $MmNi_5$ ,  $MmNi_{4.2}Al_{0.8}$ ,  $MmNi_{3.5}Co_{0.7}Al_{0.8}$ , which have the hexagonal  $CaCu_5$  type structure, have been investigated. These alloys have been prepared using mechanical alloying (MA) followed by annealing. The amorphous phase forms directly from the starting mixture of the elements, without formation of other phases. Heating the MA samples at 1070 K for 1 h resulted in the creation of ordered alloys. Gas phase and electrochemical hydrogen sorption in polycrystalline as well nanocrystalline materials have been investigated as well. Annealed nanocrystalline powders have greater capacities than the amorphous parent materials.

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## Nanocomposite permanent magnets

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Nanocomposite two-phase  $\text{Nd}_2(\text{Fe}, \text{Co}, \text{Zr})_{14}\text{B}/\alpha\text{-Fe}$  magnets have been produced from high-energy ball-milled (HEBM) and heat treated materials. Hot pressing of  $\text{Nd}_{12.6}\text{Fe}_{69.8-x}\text{Co}_{11.6}\text{Zr}_x\text{B}_6/\alpha\text{-Fe}$  composite powder containing 10 vol. % or 37.5 vol. % of magnetically soft  $\alpha\text{-Fe}$  resulted in good isotropic permanent magnets. Partial replacement of Fe by Zr in  $\text{Nd}_{12.6}\text{Fe}_{69.8-x}\text{Co}_{11.6}\text{Zr}_x\text{B}_6/\alpha\text{-Fe}$  magnets substantially reduces the temperature coefficients of remanence  $\alpha(J_r)$  and coercivity  $\beta(J_c)$  in comparison to sintered Nd-Fe-B magnets. Generally, if the content of soft magnetic phase  $\alpha\text{-Fe}$  in  $\text{Nd}_2(\text{Fe}, \text{Co}, \text{Zr})_{14}\text{B}/\alpha\text{-Fe}$  composites increases and the Nd content decreases, the thermal stability of the remanence and the coercivity increases. The value of  $\alpha$  and  $\beta$  are smaller than those of sintered Nd-Fe-B magnets.

$\text{Nd}_{12.6}(\text{Fe}, \text{Co}, \text{Zr})_{81.4}\text{B}_6/\alpha\text{-Fe}$  magnets with better temperature stability are produced due to the disappearance of the Nd-rich grain boundary phase in  $\text{Nd}_2(\text{Fe}, \text{Co})_{14}\text{B}/\alpha\text{-Fe}$  materials. A good corrosion resistance is expected, too. The oxidation behaviour of nanocomposite  $\text{Nd}_{12.6}(\text{Fe}, \text{Co}, \text{Zr})_{81.4}\text{B}_6/\alpha\text{-Fe}$  studied by Mössbauer spectrometry [1] is slower in comparison to that of the  $\text{Nd}_2\text{Fe}_{14}\text{B}$  phase in Nd-Fe-B sintered magnets. Our powder being constituted of aggregates of  $\text{Nd}_2(\text{Fe}, \text{Co}, \text{Zr})_{14}\text{B}$  and  $\alpha\text{-Fe}(\text{Co})$  grains on  $\alpha$  nanometer scale. The slowing of the dissociation process suggested, that in the aggregates the  $\alpha\text{-Fe}(\text{Co})$  phase acts as  $\alpha$  protective layer for the  $\text{Nd}_2(\text{Fe}, \text{Co}, \text{Zr})_{14}\text{B}$  nanograins against the oxidation. Goll and Kronmüller [2] drew attention to the important role of the  $\alpha\text{-Fe}$  phase in nanocrystalline  $\text{Pr}_2\text{Fe}_{14}\text{B}/\alpha\text{-Fe}$  two-phase magnets with enhanced remanence. For their samples the latter authors report that with increasing  $\alpha\text{-Fe}$  content in this composite the thermal stability of the remanence ( $\alpha$ ) and of the coercivity ( $\beta$ ) is better. The same behaviour was observed in the present work.

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## Sol-gel pastes for conductive fine line printing

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A new sol-gel printing binder based on inorganic-organic composite sol-gel materials has been developed. An epoxy-modified silane as matrix material for screen printing pastes has been synthesized. Thixotropic rheology which is necessary for screen printing has been adapted using different kinds of cellulose compounds. Several metal powders like silver, nickel or tungsten has been incorporated to obtain pastes for fine line printing. Ag containing printing systems were applied on both tin oxide coated glass and float glass using a stainless steel printing screen of 325 mesh down to 80  $\mu\text{m}$  in width with a height of up to 25  $\mu\text{m}$  after densification at 500 °C. The adhesion of the printed lines is good according to cross cut tests (class 0). Both acid (pH = 2) and basic expositions did not result in any delamination of printed lines. The electrical resistivity of silver lines with 100  $\mu\text{m}$  in width was determined to  $3,5 \cdot 10^{-6} \cdot \text{cm}$  comparable glass frit bonded fine lines. In opposition to glass frits, however, the profile of the lines as obtained by the printing step, does not change during sintering and for this reason, high profiles are obtained. This is due to the non-viscous flow sintering based on  $\text{SiO}_2$  nanoparticles, which densify far below  $T_g$  at 500 °C.



## Micromechatronics - a foundation for future products

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During the past years system thinking and interdisciplinary cooperation have found increasing influence in engineering. They are now the foundation for emerging new interdisciplinary research directions e.g. Mechatronics, Bionics, Medical Technology) and new courses that are well accepted by students.

Micromechatronic products are characterised not only by the interaction of mechanical, electrical, electronic and control functional elements but also by components whose function is based on the operation of microsystem structures.

The new quality in comparison to classical mechatronic products is achieved because the design and miniaturisation opportunities of the microtechnologies can consistently be utilised for the miniaturisation and spatial integration. In the microsystem domain even complex components can be manufactured cost-effectively using batch-processing.

Micromechatronic products are characterised by the following features:

- Increased degree of integration for systems with active mechanical components,
- utilisation of functional principles and structures up to their physical limits based on powerful control (technical intelligence),
- extended functionality through the implementation of auxiliary functions important to the application,
- increase in efficiency, flexibility and dramatic reduction in volume.

Due to the permanent increase in complexity and heterogeneity of micromechatronic systems their development can be done only with suitable design methods on powerful computer platforms using synthesis friendly software. This allows to achieve a global optimum for the whole system under the given customer requirements. On this basis it is possible to e.g. design micromechatronic actuators that comprise a large number of coupled single actuators. As with biological motion systems the external motion behaviour is generated from the interaction of the single actuators which allows to change its motion style, order and degree of freedom by control of the single actuators.

The trend towards miniaturisation of functional modules as components of multifunctional systems leads to a complexity like that of biological systems. Mechatronics - as distinguished science subject still in its explanatory stage - can be much more open to bionic influences than traditional domains of science since by integrating unconventional technologies, new materials and designs it likewise particularly requires integrative thinking. To control the effects of the complexity of its products like those emergent (not being based on the single element) and nonintentional (risks and side effects) functions increasingly requires the knowledge of the nonlinear behaviour of such highly complex systems like they are investigated in Biology.

It is shown how stimulation for the design of new technical motion systems can be

drawn from the analysis of selected biological motion systems.

In this context demands are created for the development of new materials in particular for an effective energy conversion of the mechanical energy required for the motion and for the integration of single actuators or actuator components. Beside the application of solid body effects for the mechanical energy conversion in particular those materials are of interest whose transmissive function is based rather on the molecule design i.e. its texture than on its external geometry. Materials whose mechanical properties are controlled locally and reversibly and whose intrinsic sensor effect provides shape control or whose shaping process under functional stress is controlled in a closed loop will not be reserved to living nature anymore.

By means of examples cascaded drive systems are presented whose mechanical connection is tuneable and where activation of single elements is possible in principle. This not only multiplies the external motion variable but creates an opportunity for adaptive behaviour.

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## A new method of quantitative evaluation for the adhesive toughness of thin films and substrates

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Adhesion is one of the critical problems for thin film structures, which is quite difficult to evaluate. In this paper, a new method of quantitative evaluation is reported for the adhesive toughness between thin film and substrate, which gives an absolute measure of adhesion in terms of macroscopic mechanical energy required to extend interface crack of unit area. In this method, the cross section of film and substrate is formed where the film is projected a little out of the edge of the substrate. Interface crack is initiated and extended by applying external load to the projection of the film from interface side with a diamond needle. In this process, the applied load is monitored as a function of interface crack extension which is observed by an optical microscope. On the other hand, the load required to extend the interface crack is numerically calculated also as a function of crack extension with an assumed value of toughness at the interface crack tip. By comparing the experimental data with the results of numerical simulation, the adhesive toughness can be evaluated. As an example of application, we evaluated the adhesive toughness between the chemical vapor deposited thin diamond film and the Co-cemented tungsten carbide substrate.

## X-ray texture and residual stress analysis on photoactivated electroless plated Cu-layers

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Photoactivated electroless plating by means of incoherent light is an advantageous method for the selective copper metallization of dielectric surfaces. Using this method a structured seed layer is manufactured by patterning an activated film of Palladium acetate with the standard photolithographic method. After reducing the  $\text{Pd}^{2+}$ , the resulting  $\text{Pd}^0$  seeds are able to catalyze many metals from suitable commercially available electroless baths. However, the bonding strength of the deposited structures strongly depends on the deposition conditions, and under non-ideal conditions the layers fail by peeling off. Pronounced intrinsic stresses are assumed to be a reason for this behaviour. Thus, an optimization of the electroless deposition process requires an enhanced understanding of residual stresses and textures in the manufactured layers.

Therefore, in this paper the development of residual stresses and texture in Cu-layers was investigated exemplarily using X-ray diffraction. For the manufacturing of homogeneous layers with large lateral dimensions a sputtered seed layer of Au was employed instead of the structured Pd seed layer. Onto this homogeneous seed layer, the Cu was deposited electrolessly using two different commercial baths. The thickness of the resulting Cu layers ranges from 0.33  $\mu\text{m}$  to 4.6  $\mu\text{m}$ .

Using the  $\sin^2\psi$ -method always rotational symmetrical tensile residual stress states were determined which were in the range of 20 to 250 MPa. These tensile residual stress could be assumed to be a reason for a failure by cracking but not for the observed failure by peeling off. According to that, the layer failed had a comparable small tensile stress state.

The texture development was characterized using orientation distribution functions (ODF) which were calculated from pole figures of the diffracted X-ray intensity. Fibre textures were found in all layers with fibre axes varying strongly as a function of the layer thickness and the bath used for deposition. A slight correlation between the intensity of the texture components and the failure by peeling off was found. The influence of the sputtering texture of the seed layer was also discussed.

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## Residual stress determination in microsystems using X-ray diffraction

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The trend in microsystem packaging towards more compact structures gives rise to numerous problems of mechanical reliability. The mechanical reliability of microsystems is a very difficult field with a lot of open questions depending on structure size and material behavior of the various joined components.

In this situation, the knowledge about the stress state and its variations, regarding the different steps of the manufacturing process and under in-operation conditions, is necessary to obtain optimum properties as well as to avoid failure and damage to microsystems and microdevices.

X-ray diffraction is a powerful tool in the investigation of residual stress states in microsystems, because it permits very small measures of volumes to be examined without destroying them. As the only requirement for the X-ray diffraction method is a crystalline state of the specimen, the examination of a wide range of materials and components becomes possible.

Due to the small thickness of the individual layer in multi-layered microsystems, the X-rays penetrate the samples deeply in many cases, and so diffraction patterns of layers of different materials can be examined.

Some examples should illustrate the broad field of X-ray residual stress investigations in microsystem techniques.

For the successful execution of X-ray residual stress analyses on microsystems, some special conditions must be fulfilled:

- For the measurement of details diffraction equipments like Eulerian cradles with sample holders for the realization of a wide range of x, y, z-measuring positions have proved suitable.
  - The miniature size of the measurement spot inevitably leads to a loss of radiation intensity and prolongation of exposure time for the diffraction procedure. This disadvantage may be compensated by using powerful radiation sources.
  - Another solution to this problem would be the application of improved X-ray optics like mirrors, lenses and capillaries in the path of the diffraction beam.
  - A third way is the use of new diffraction systems with area detectors.
- Using new X-ray optics like fibres and mirrors, the size of the measurement spots can

be reduced up to very small lateral dimensions without unacceptable losses of intensity. As a result of this miniaturisation the material within the measurement volume more and more differs from the polycrystalline state. In this situation new detection systems like area detectors can be used successfully and new evaluation procedures are required.

## Metal recycling as renewable resource of clean energy and raw materials

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Nowadays mankind is confronted with the fact of living in a closed environmental system; having only one input materialized in the dwindling natural resources, and only one output spotlighted in the enormous amount of waste from the human production/consumption activities. This fact emphasises the finite nature of the globe and expresses the laws of conserving energy and mass in a global way.

The exhaustion of natural resources and environmental awareness highlighted the necessity of waste recycling all over the world. The present paper deals with the benefits of metal recycling with respect to the Trio of energy, environment and economy.

Practical examples concerned with the production of ferrous sintered compacts, Aluminium / Silicon alloys, Aluminium Bronzes, ...etc from industrial wastes are given and discussed.

## Micro- and nanostructures – preparation and applications

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In our global society, communication and information transfer is playing a role of exponentially increasing importance. This means that there is always a need for increased, faster information transfer. Since the velocity (the distance divided by time) of the information transfer is limited by the speed of light, the only real strategy for improved throughput is to make the distance and in turn the physical structures smaller. This need for ever increasing information processing speeds has driven the ever decreasing structure sizes applied in microelectronics. A new challenge exists, namely to exploit the very successful microelectronics technology to create and realize quite new devices and entirely new micro- and nano-systems.

There are a number of corresponding technological and material problems hindering the proliferation of these small systems. Nanometer-scale structures which actuate or act over a short, sub-picosecond period demand a better understanding of material properties on these same scales and therefore necessitate the development of new material evaluation schemes. Based on this improved understanding of essentially molecular scale material properties, drastic improvements and quite new materials may be realized, and from this new technological possibilities and even smaller structures can be developed. This would necessitate further refinement in material understanding, in essentially a self-sustaining iterative process.

To realize the smaller structures, they must be written in a sensitive resist and then transferred into the corresponding substrate. Therefore, one must always deal with lithography and dry etching problems. In this paper, we will concentrate on the main problems of lithography and dry-etching and present new applications in the form of micro- and nanosystems (MEMS) and especially those used as sensors for scanning probe microscopy.



## Fritting contacts utilized for micromachined wafer probe cards

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We have investigated the characteristic of the fritting which should be utilized for making contact to IC pads in micromachined wafer probe cards. Micromachined probe cards made on Si substrates have some advantages over conventional needle ones. They can be applied to higher pad-density and smaller pad-pitch chips, and can be effective in the tests using high-speed signals above 1 GHz, since the dimensions of micromachined structures can easily be in the range of a few tens of micrometers. Moreover, there is no problem of thermal expansion mismatch between the probe card substrate and the wafer under test. Additionally electrical circuits for testing can be integrated into the substrate and actuators or sensors can be built into the probes. If a probe card consists of an array of actuator-integrated microprobes, it gains some further advantages. Since the deflection of each probe can individually be controlled, probe-pad contact force can be uniform by compensating the probe-pad distance deviation with probe deflection. Further, since contacts can directly be switched on and off, it could be suitable for a wafer level test/burn-in probe card. The critical problem of the micromachined probe cards is that each micromachined probe can not produce or endure the force required to break the oxide on Al pad surface mechanically, which is commonly reported to be more than 100 mN. However, as Bailey et al. showed, the mechanical scrubbing motion can be replaced with the electrical breakdown of the interfacial oxide, which is known as fritting.

In this report, we measured the forces necessary for making contact to Al pads, and for disconnecting the contact, when using the fritting process. Electroplated Au bumps and Ni bumps are used as the contact probes. As a result, the fritting is able to get low resistance contact to Al pads without applying external forces. The adhesion force required disconnecting the contact between the Au bumps and Al pads were measured to be 0.4 mN, being one third those when using Ni bumps. The force in this magnitude range is small enough for micromachined cantilever-type active probe to generate. This indicates that we can design a microprobe for micromachined switching probe cards. We will also report on the investigation of the fritting process when using gold-coated AFM cantilevers as the contact probes to study the influences of the shapes of the probe tip on the adhesion force and contact resistance.

## Finite-Element-investigations on testing devices for solder joint reliability evaluation

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The entire strain in experiments to determine the creep behavior of solder joint materials is a combination of elastic, inelastic, plastic and creep deformation. Most of the fatigue tests currently are performed using strain control. The separation of the creep fraction of the whole deformation, which is essential for life time prediction, is difficult in strain controlled experiments. Using load control instead it is relatively easy to extract the accumulated deformations relevant for solder joint damage. Cyclic tests have to be used to determine the amount of creep deformation causing damage of the solder joint. The enveloping strain curve, which is showing primary, secondary and tertiary sections, respectively, like creep curves in static experiments are used for determination of irreversible strains during loading and dwell periods. Because the secondary time period is dominating in comparison to the primary and tertiary ones, the strain rate during secondary creep is used for material characterization under certain load and temperature conditions. The relation between the accumulated creep strain per cycle calculated from the average secondary strain rate multiplied by the cycle duration and the performed cycles to failure usually is expressed in Coffin-Manson-type equations for solder materials.

The characterization of the mechanical properties of solder materials, especially of stress-strain relations and fatigue resistance is performed using shear test equipment, because the major loading of solder joints is consisting of shear straining. To evaluate quantitative relations a homogeneous distribution of shear stresses is necessary. To compare different shear tester geometries the stress distributions have been determined using Finite-Element models of Single-Lap-Test, Double-Lap-Test, Ring-and-Plug-Test and Iosipescu-Test and different solder compositions.

**Keywords:** Material characterization, finite element investigations, solder joint reliability

## Thermo-electro-mechanical analysis of piezoelectric micro-components by harmonic oscillations

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The microsystems are widely used in precision mechanics, optics, information and communication systems, medicine and bioengineering. Different actuating principles for the microsystems are known such as piezoelectrical, electrodynamical, thermal and so on. The piezoelectric actuating principle are widely used because of its small power consumption and high efficiency in microrange.

Different constructions of piezo-microcomponents are used for actuation of the microsystems. The monomorph piezoelements can be used as an actuator in micropositioning and microgrippers systems. As an actuating element in micropumps and microdosing systems a bimorph piezoelectric membrane is preferable.

All this microsystems can be actuated statically or dynamically. The result of the actuation at the dynamic mode with high frequencies is a self-heating of the piezocomponents through the internal viscous friction. The characteristics of the microsystems in great extent depends on the temperature influence of the thermal fields. It should be taken into account while designing of a whole microsystems construction.

The paper presents an approach for the thermo-electro-mechanical analysis of the piezo-microcomponents. The problem can be solved through a following algorithm,

- solving of the problem of mechanical vibrations: calculation of the natural modes, frequencies and forced harmonic oscillations,
- calculation of the energy dissipated through mechanical vibrations into the piezomaterial,
- solving of the problem of nonsteady thermoconductivity with dissipation function as a source of thermal energy,
- correction of the piezomaterials electromechanical coefficients in dependence on calculated thermal fields,
- determination of the piezo-microcomponents characteristics.

This analysis makes possible to determine the following parameters of the piezo-microcomponents:

- the self-heating temperature fields,
- the dependencies of the natural frequencies and deformations on temperature,
- the amplitude-frequency response with thermal influences taken into account.

It has been shown that the errors from temperature deformations reach the order of deformations of electrically actuated piezo-microcomponents and should be corrected during their control.

## Combination of electroless and electroplating methods in electronics and instrument-making

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The objectives of this work were the development of new, competitive technologies allowing us: to exclude disadvantages of metallization methods existing so far; to regulate physical-chemical properties of the coatings in the wide range; to obtain coatings with given properties; to replace adequately noble metals by non-noble ones; to exclude toxic substances [1-3].

For the development of an optimal technology every step of the composition of solutions and regimes of electroless deposition, conditions process: preliminary treatment of various substrates (The sensitization and activation of photolithography, selective etching processes, etc. were improved. For obtaining coatings with the given properties (both on bulk and powder-like materials – in particular ceramics, carbides, nitrides, oxides, zeolits, diamond, etc.) the influence of different factors, such as temperature, concentration, pH, additives, surface roughness, etc. on sensitization, activation and electroless deposition have been investigated. An activation mechanism of dielectrics was established.

The developed technology of modification of various materials in the work allows to use it for obtaining: high-quality adsorbents; catalysts and powder-like metallized materials; current-conducting lines (on dielectrics, semiconductors and plastics); ohmic contacts; diffusion barriers; vacuum – proof soldering; increase of microhardness, wear-, temperature- and corrosion resistance; for shielding electronic devices against electromagnetic interference.

The new competitive technologies for microminiaturization, as well as a unique design for production of two-layer photomask with semitransparent masking elements by means of single, conventional photolithography was developed. The developed methods enable us to solve one of the main problems in modern microelectronics.

The proposed new methods allow us to produce, for the first time, microdevices with adjacent elements made of various materials of different thickness by single photolithography on the same substrate. These advantages increase the possibilities for device designs and their functional purpose and simplify the removal of undesirable gases and heat dissipation.

The new methods and device of a new design for precise measurements of ductility by means of bending are presented.

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## Electroless metallization of non-metallic materials

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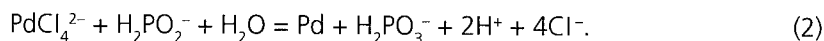
The results of systematic investigations of the mechanism and kinetics of the sensitization and activation have been described [1-3]. The influence of temperature, time, concentration, pH additives on Sn, Pd ions adsorption and electroless deposition were investigated by radioactive isotopes, XPS, electron microscopic and photometry methods.

The obtained data illustrates the influence of substrate roughness on the surface concentrations of Pd and Sn. It is clearly observed that with the increase in substrate roughness (ground surface), concentrations of these elements also increase.

A part of palladium ions, not reduced by sensitization:



can be partially reduced at the subsequent interaction with hypophosphite according to the reaction:



The developed methods of metallization enable us to obtain pore-free, thin and thick coatings of uniform thickness having low internal stress and high adhesion on complex-shaped metallic, non-organic dielectric, plastic and semiconducting materials with any surface roughness including polished surfaces. These methods give us a possibility to regulate physical-chemical properties of the coatings (conductivity, chemical, mechanical, optical and magnetic properties, etc.) in the wide range and to extend significantly the function of devices made of dielectrics, semiconductors, plastics and metals. As a result of implementing the developed methods of modification and metallization of non-metallic materials in mass production of quartz resonators and filters, monolithic piezoquartz filters, piezoceramic articles for hydroacoustics and delay lines of color TV sets, photomasks, ICs, resistors, capacitors, etc. Au and Ag are adequately replaced by non-noble metal alloys, toxic substances are excluded, the processing time for making devices are sharply reduced and the technology is significantly simplified. The proposed new methods allow us to produce for the first time microdevices with adjacent elements made of various materials of different thickness by single lithography on the same substrate.

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## Modelling the cure-dependent viscoelastic behaviour of a thermosetting resin and residual curing stresses

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Thermosetting resins, like unsaturated polyester, are widely used as the matrix for structural composite materials. A significant volumetric shrinkage occurs during the curing (or polymerization) process of these resins, which is due to the formation of a crosslinked network from the polymer chains. Curing shrinkage will affect the quality and the mechanical performance of the produced composite parts by inducing residual stresses in the material and consequently warpage and microcracking. In order to improve the knowledge on the curing effects, this research is devoted to modelling the development of curing stresses in the resin and resin-based composites.

During the curing reaction, the liquid resin transforms to a gel, rubber, and finally to a viscoelastic solid material. Simultaneously, the curing shrinkage occurs which results in curing stresses after the gel point of the resin, if the resin is somehow constrained e.g. by a surrounding mould, by a stiff substrate, or by internal reinforcing fibres. On the other hand, the development of the curing stresses is partly reversed by stress relaxation due to the viscoelastic behaviour of the curing resin. In the present work, a cure-dependent viscoelastic model is proposed for the curing resin, for which the material parameters are determined through rheological tests called dynamic mechanical analysis (DMA). An incremental constitutive equation is developed and implemented in a finite element code in order to analyse any three-dimensional stress field. The input to this model is the curing shrinkage of the resin, in addition to the cure-dependent material parameters, which is also measured in a designed equipment. The modelling results for the curing stresses and deformations are verified by the results of experiments in which the resin is constrained in one, two, or three dimensions. In this work, the shrinkage effects in composites are also investigated from both micromechanical and macromechanical points of view.

## Ion assisted growth of ultrathin diffusion barriers on low dielectric constant polymers

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TiN and TaN thin films are proposed as barrier layers between copper interconnects and low dielectric constant (low-k) polymers. As the thickness of the barrier layer is scaled down, the nitride-polymer interface will become an important issue for the effectiveness of the barrier and the reliability of the interconnect structure. In order to evaluate nitride formation and the interfacial chemistry we deposited TiN and TaN on fully cured low-k polymers by two different techniques: reactive evaporation of the metal in nitrogen ambient and by ion assisted reactive deposition using a low energy (0.1 - 1 keV) nitrogen ion beam during evaporation. Photoelectron spectra were recorded in situ for metal coverages from 0.1nm until bulk like metal or metal nitride spectra were obtained. Plain view TEM was used to image the morphology of selected films. Nitride concentrations, extracted from the photoelectron spectra, show that even though very similar nitride films are produced by both techniques for thicker films (>5nm) we only find significant amounts of nitride at the interface in the ion assisted case. The interfacial chemistry of the reactively grown TiN and TaN is very similar to the growth of the pure metals. In both cases we observe the formation of compounds with carbon and oxygen from the polymer rather than the formation of nitride. In contrast, the use of low-energy ions increases the amount of nitrogen incorporation at and near the interface, resulting in a much more homogeneous film.

Ion beam damage to the polymeric substrates and ion induced intermixing was investigated by angle resolved photoemission at different ion energies. Optimization of the ion energy seems to be necessary to minimize ion beam damage or nitrogen incorporation into the dielectric. Significant ion induced intermixing at the nitride polymer interface only occurs at higher (1keV) ion energies and is of less concern for the heavier metal Ta. In summary it was found, that ion-assisted deposition with carefully optimized parameters can significantly improve the homogeneity of nitride barrier layers for copper based high density interconnects.

## Investigation of interface cracking in electronic packages

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Cracking in various interfaces is a significant concern for yield loss and reliability of advanced packages. There are many reasons for interface damage, for example, low adhesion due to incompatible interfaces or due to contamination. Delamination at the encapsulant-substrate or chip-substrate interfaces can lead to cracking of the electrical interconnects.

To assess the integrity of the interface, the following information is necessary: the interfacial adhesion, a pertinent simulation model, and an estimate of the maximum length of a permissible initial crack.

In this paper a method is described to investigate the interface strength and the process of delamination and to pursue crack initiation and crack propagation. The 3-point-bending-test is combined with the optical measuring procedure **DAC** (**D**eformation **A**nalysis by **C**orrelation), a method of digital image processing. Digitized images of the specimen under different bending load steps are compared by a correlation algorithm. This allows to determine and to evaluate the local displacements and displacement fields.

Another interesting combination of the DAC method is the very precise determination of the time and location of beginning of delamination.

The investigations of local displacements and displacement fields by optical methods in combination with a Finite Element Analysis lead to a better understanding of crack initiation and crack propagation and consequently to detailed knowledge about package stability.

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## Real-time monitoring and measurement of mechanical properties of polymeric films using resonant string structures

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Understanding mechanical properties of polymeric thin films is very important in manufacturing and reliable operation of the devices employing polymers. Two in-situ measurement schemes, using resonant string structures, for the measurement of the polyimide residual stress and polyimide/metal adhesion durability have been developed. A thin film residual stress measurement scheme employing Rayleigh's method is proposed. According to the Rayleigh's method, the resonant frequency of a polyimide string can be related to the film stress. By measuring the resonant frequency of the polyimide strings, fabricated using a bulk-micromachining technique, the residual stresses have been calculated. The measurement results have been compared with various conventional measurement techniques for verification.

Also, a scheme to quantize the adhesion durability between a polyimide film and a metal film has been developed. This scheme is based on a polyimide/metal bimorph string structures, fabricated using a surface micromachining technique, vibrating with an alternating potential. The change of resonance profile of this string structure can be related to the degradation of adhesion strength at the polyimide/metal interface. Various polyimide/gold string structures have been fabricated and the change of resonant qualities has been monitored over a time period. Notable changes of resonant Q-factor and resonant frequency, due to the degradation of adhesion between the metal and polyimide, have been observed after  $10^8$  cycles (string vibration) for the polyimide/gold bimorph strings.

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## Nondestructive evaluation of fracture characteristics by quantification of Barkhausen noise method and microstructure analysis

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Barkhausen noise method as the magnetic nondestructive test have the advantages for evaluating the properties of magnetic material precisely and high-sensitively compared with other magnetic NDT methods. For a long time Barkhausen noise method can be applied to measure the bulk magnetic properties of magnetic materials and recently to evaluate microstructure, stress analysis, fatigue, creep, and fracture characteristics as a NDT method. But so far Barkhausen noise method has been used as evaluating trends of material properties qualitatively rather than quantitatively. For this reason, many NDT testing methods have scarcely been applied to industrial plants and laboratory. In this investigation we make experiments on the variation of Barkhausen noise as microstructure, and quantify Barkhausen noise(voltage) via formula of velocity of magnetic domain walls using coercive force as retarding force of domain wall movement. As a result, we can evaluate the microstructure of magnetic materials and trends of fracture toughness quantitatively by measuring Barkhausen noise. Therefore we can directly evaluate microstructure and fracture toughness by Barkhausen noise method as accurate in-situ nondestructive testing method.

## Effect of intermetallic compound layer development on mechanical strength of 63Sn-37Pb solder joints

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Metallurgical interactions occur between the solder and other materials by means of solid state processes and the resulting reaction products (intermetallic compounds) will continue to grow as the solder joint ages. For example, in Cu/63Sn-37Pb solder joints, the copper-rich intermetallic  $\text{Cu}_3\text{Sn}_5$  forms adjacent to the copper and the intermetallic  $\text{Cu}_6\text{Sn}_5$  forms adjacent to the solder. Formation of these intermetallic compounds affects the mechanical integrity of solder joints since the solder cracks tend to be generated near the intermetallic compound. The present paper examines the tensile strength of the solder joints after thermal aging. The specimen consists of two FR-4 plates with 1.7mm thickness, plating layers and 63Sn-37Pb solder joint. As to the plating metals, Cu and Ni were employed. The specimens were placed in an isothermal chamber with 170 degrees centigrade for one to 80 days. After cooling down for one day at room temperature, tensile tests were performed. The tensile load was applied perpendicular to the solder joint layer with cross head speed of 0.5mm/min at 22 degrees centigrade. The load increased linearly with displacement and specimen broke suddenly at the maximum load. It was found that interfacial strength is higher for Cu/63Sn-37Pb system than for Ni/63Sn-37Pb system. By fractographic analysis using scanning electron-microscope, dimple type fracture was observed for Cu and 63Sn-37Pb interface while the cleavage type fracture for Ni and 63Sn-37Pb interface. The interfacial strengths of both systems decrease with aging period firstly, while they become almost constant if the aging period is over 30 days. Thickness of intermetallic compound of Cu/63Sn-37Pb system ( $\text{Cu}_3\text{Sn}_5$  and  $\text{Cu}_6\text{Sn}_5$ ) and Ni/63Sn-37Pb system ( $\text{Ni}_3\text{Sn}_4$ ) increases in proportional to the square of aging period and it is much thicker for Cu/63Sn-37Pb system. These results suggest that the interfacial strength depends strongly on the mechanical properties of intermetallic compounds generated, and the effects of the intermetallic compounds on the interfacial strength become a limit state when their thickness exceed a certain critical value.

## Microstructure characterization and mechanical properties of Mo-Cu composite for electronic packaging applications

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In recent years, the Mo-Cu composite as a heat sink material for integrated circuits has been developed because of its relatively low thermal expansion coefficient and high thermal conductivity. Starting from the powder, the feedstock is manufactured through a metal injection moulding (MIM) process. After MIM, a thermal debinding and a liquid phase sintering process is applied. In this paper, the Mo-Cu composite was characterized through scanning electron microscopy and atomic force microscopy topographically, and through nanoindentation mechanically, whereas hardness and Young's modulus were determined. The materials under investigation have been provided by Dr. K.-H. Wichmann and A. Jung, DASA Ulm.

## Mechanical properties of micro-injection moulded components

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Micro-injection moulding is a method to produce small polymeric components. How the processing conditions influence the materials properties is an actual question.

The results of micro-injection moulding are parts having a mass of only a few milligrams. Due to this fact it is obvious that conventional material testing methods can not be used. The instrumented nano (and micro) indentation test was used for the mechanical characterization of such small parts and components. From load - penetration depth curves hardness and modulus can be calculated. The measurements were done on injection moulded films and components using a nanoindenter.

The films were test specimens with dimensions of 10 x 5 mm and thicknesses of 100, 50 and 30  $\mu\text{m}$ . The films can be used to optimize the processing conditions. Measurements were done on the surface and over the cross section of the specimens. Reducing the thickness of polypropylene (PP) and polyoxymethylene (POM) films to 50 and 30  $\mu\text{m}$  do not result in visible changes of hardness and modulus. Also deviations of hardness and modulus in dependence on measurement positions on the surface are small. Indentation measurement of liquid crystalline polymers (LCP) is difficult because of the strong orientations, which are results of the typical structure of LCPs. Both measurement parallel and perpendicular to the injection direction lead to very large standard deviations. Bending of small beams using the indentation apparatus is a possibility to solve the problems of modulus determination. The beams were cut from the films and one end of them were fixed on the specimen holder. So it acts as a cantilever beam. The determined value of modulus of elasticity (7500 MPa) is comparable to values measured by dynamic-mechanical analysis in tensile mode but low compared to the Young's modulus of the bulk material (11000 MPa).

Miniaturized gear wheels, for example applied in watch industry, are »classical« micro-injection moulded components. To look if the materials properties are kept in the small parts measurements were done on the cross section of some teeth of such gear-wheels (material: POM). The modulus (about 3000 MPa) and the hardness (about 220 MPa) were in good agreement with the values of the bulk material.

Other examples of small components are cases of hearing aid sensors also made of POM. At present two case forms are discussed, one with relatively sharp and one with relatively blunt radiuses. In both variants the properties of POM are at the same level like in the bulk material.

By means of the presented results the suitability of instrumented indentation test for testing of micromoulded parts can be proved.

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## Properties, characterisation and challenges of semiconducting polymer applications

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Semiconducting polymers combine the strong processability advantages of polymers with electronic activity. A large variety of optoelectronic devices such as photodetectors / solar cells, light-emitting diodes (LEDs), field-effect transistors (FETs), microcavities, lasers and simple integrated devices have been fabricated from semiconducting polymers. Diode structures are easily fabricated by spin-coating the polymer from solution onto a substrate that is covered with an transparent electrode, and thermally evaporating the top electrode onto it. The technological development and commercial exploitation of semiconducting polymer devices requires a sound understanding of the underlying photophysical processes. Semiconducting polymers consist of alternating carbon-carbon single and double bonds. The resulting 1-dimensional delocalised  $\pi$ -electron system gives rise to the semiconducting nature of the polymers.

When a voltage is applied across the polymer film, spin-+ electrons and holes are injected from the electrodes into the polymer. They migrate along the polymer chains and capture to form a strongly bound electron-hole pair termed exciton with an overall spin of 0 (singlet exciton) or 1 (triplet exciton). Singlet excitons are emissive, but emission from triplet exciton is spin-forbidden. However, since statistically 3 times more triplet excitons than singlet excitons should be formed in a polymer LED, a theoretical efficiency limit for polymer LEDs of 25 % is given. We were able to open a pathway to overcome this theoretical limit by harvesting emission originating from both singlet and triplet excitons. To do this we added a platinum porphyrin as a »dopant« to a semiconducting polymer host. The platinum in the porphyrin is a heavy metal and thus induces a strong spin-orbit coupling, which renders triplet emission (phosphorescence) allowed. Singlet and triplet excitons created on the polymer are transferred to the porphyrin, where both species can emit.

For such device engineering, it is crucial to know the energy of the triplet level in semiconducting polymers. We have synthesised semiconducting polymers with platinum incorporated in the polymer backbone and compare their electronic structure to analogue semiconducting polymer without platinum. From this we are able to deduce the energy levels of the triplet states in the purely organic compounds.

Optically pumped semiconducting polymer lasers have been demonstrated. To explore the avenue towards electrically pumped polymer lasers, we have investigated the response of LEDs to pulsed voltage excitation, thus reaching high excitation densities while avoiding excessive device heating.

Field-effect mobilities in polymer FETs of  $0.1 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$  were achieved by using self-organisation of suitable polymers. With these we were able to construct a simple integrated device consisting of a polymer FET driving a polymer LED.

In photocells made from semiconducting polymers, photogenerated excitons are dissociated by electron transfer at the interface of donor and acceptor polymers. To obtain a large polymer-polymer interface combined with a good connectivity of the respective polymer to the collecting electrodes, we developed a two-layer diode structure, fabricated by a lamination technique followed by controlled annealing. Short-circuit quantum efficiency of 29% and overall power conversion efficiency of 1.9% are reached with this structure, demonstrating that there is a promising potential for practical applications, considering the ease of processing.

## Development and applications of sensors for deep space explorations

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Sensors have wide applications in planetary explorations; they have been and will be used to monitor spacecraft equipment performance, in guidance and navigation, for planetary seismology, meteorology, planetary atmospheric chemistry including search for life, as well as for in-situ analysis of samples. Large advances in sensors performance and miniaturization have made possible to develop concepts of micro and nano sciencecraft. However, much work remains to be done. The space environment is harsh: extreme heat and cold, thermal cycling, radiation effects, and corrosive environments put conventional sensor device designs at risk. Reliability, packaging and flight qualification methodologies need to be developed for all sensors to produce robust devices for a successful future in space missions. These issues are discussed in the paper, along with examples of new types of sensors, MEMS based sensors, and sensor integration to enable System-On-A-Chip (SOAC) and lab-on-a-chip.

## Characterization of gated p-i-p-structures on CVD diamond films and natural crystals

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Diamond CVD films and natural crystals were doped with boron using multi-energy implantations of energies and doses ranging from 36 keV to 95 keV and  $2 \times 10^{15} \text{ cm}^{-2}$  to  $3 \times 10^{15} \text{ cm}^{-2}$  to manufacture p<sup>+</sup>-i-p<sup>+</sup>- and p<sup>+</sup>-p-p<sup>+</sup> electronic structures. These devices operate as enhancement transistors. The i-gaps range from 2.5  $\mu\text{m}$  to 18  $\mu\text{m}$  with a length of 100  $\mu\text{m}$ .

These samples were first optically characterized using cathodoluminescence-, Raman- and photoluminescence spectroscopy at room temperature and at 77K. The quality of the CVD films and the natural crystals have been determined by means of to the intensities and FWHM of the diamond Raman peaks and the free exciton lines. The measurements of the nitrogen (H3, 575 nm) and silicon (738 nm) centers yielded additional valuable information about the purity of the materials.

I-V-curves of the electronic structures have been measured from room temperature up to 100 °C. From these data the activation energies and the concentrations of the deep traps were calculated. From the same curves, other parameters such as the onset voltages, the currents at the onset voltages as well as the onsets of the space-charge-limited-current regimes were determined. The dependency of these parameters on the i-gap was measured and compared to the theoretical expectations and to the results of the optical measurements of the samples.

The natural crystals showed the best electrical behavior, i.e. lower onset voltages (for the p<sup>+</sup>-p-p<sup>+</sup> structure 35 V at a current of 5 mA/cm<sup>2</sup>).

## Simulation of resonant interference of light by thin films

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The phenomenon of light interference in flat-parallel media of Fabri-Peros type is used [1-3] to get dispersive relations  $n(\lambda)$  in the region of resonant transitions of exciton - polaritons in thin films by means of well-known expression

$$m\lambda_m = 2nd,$$

where maxima of reflection correspond to the whole values of  $m$ , and minima – to half – whole values of  $m$ .

In no way we want to deny validity of the above expression in the whole, but it is difficult to agree with the proposed by authors scheme of decoding the interference spectra of reflection.

In the present paper the conditions of extrema localization of light arbitrary reflection by Fabri-Pero's resonators owing to the method of reflection complex amplitude hodographs are studied. It is determined that light reflection spectrum by more dense than environment free resonator in long wave region always begins with maximum of first order  $m=1$ , and absorption in the resonator does not influent essentially on spectral position of extrema. The condition

$$m = \frac{4nd}{\lambda_m}, \quad m = \begin{cases} 1, 2, 5, \dots - \max \\ 2, 4, 6, \dots - \min \end{cases}$$

of extrema localization, than provide correct decoding of the extrema in reflection spectra and modeling the dispersive relations of polariton optical characteristics in thin films is obtained.

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## Multi-level modelling of heterogeneous micro materials

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Most of the materials utilised in industry are heterogeneous. Examples are metal alloy systems with a second phase in the form of precipitates and pores, and texture of polycrystalline materials. In case of micro materials these heterogeneities significantly change the material properties of the whole structure (such as micro part). As for the micro parts the size of the heterogeneities is only a few orders of magnitude smaller than the size of the whole structure, it is not possible to use homogenised material parameters because micro materials show strong dependence of the behaviour on a loading history as well as on the size and shape of the structure. At the same time usually it is still impossible to create a finite element mesh that both accurately represents the microstructure and allows numerical solutions within a reasonable amount of time on today's computational systems.

To overcome these obstacles the multi-level approach to homogenisation may be used. The approach presented in this work is a development of the method proposed by Smit [1]. The structure under consideration is discretised by finite elements (and further referred as macrostructure). The physical and geometrical properties of heterogeneities are identified by Representative Volume Element (RVE). For each macroscopic integration point a separate finite element computation at RVE, assigned to this integration point, is performed. The macroscopic stress is obtained by applying the macroscopic deformation on the RVE through imposing appropriate boundary conditions and averaging the resulting RVE stress field. The consistent stiffness tangent at the macroscopic integration point is derived by reducing the RVE stiffness matrix.

Applying only the deformation tensor from the macroscopic integration point on the RVE implies that (i) the size of the RVE is infinitely small in comparison with the size of the whole structure and (ii) the periodicity conditions on the RVE should be prescribed. Both of these conditions are not the case for micro materials, where the size of the heterogeneity plays role and in general every part of the microstructure deforms in a unique way. These conditions may be accounted by prescribing to the RVE not only the deformation tensor itself, but also its gradient.

The method enables the incorporation of large deformations and large rotations and suitable for arbitrary material behaviour, including physically non-linear and time-dependent.

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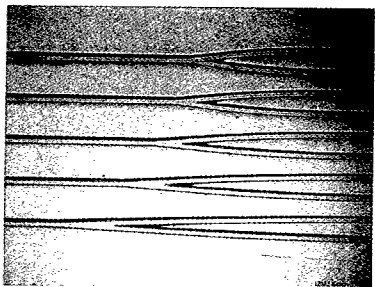
## Property profiles of materials for micro-structuring by hot-embossing

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With an increasing number of applications in the fields of micro-systems, optical telecommunication and chemical or biological micro-analytics, the need for suitable materials and optimised micro-structuring technologies grows, too. Besides present technologies like photolithography or laser micro-machining, especially new replicative processes are also used to form exact structures in special materials. This is a major point of interest for low cost aspects and a mass-market. Our paper will present aspects related to material, processing, and packaging of hot-embossed structures.

The hot-embossing process itself works in this way: While the processing tool is imprinted into the material, temperature and pressure are being increased according to a well defined regime. The tool's surface is a negative of the requested structure. After cooling down and detachment of tool and work piece, the structure is transferred to the special material.



Microscopic photograph of micro-channel splitters, channel width: 60  $\mu\text{m}$ .

According to the requirements of system application, micro-structuring process and system packaging, a definite property profile of materials is necessary. For example, optical waveguides for high-speed data-transfer on board or module level in electrical-optical systems<sup>1</sup> need a completely different property profile for the used material in comparison to micro-channels for wet chemical analytics. Different types of material should be structurable by the hot-embossing process. We will discuss the general problem of material and its structuring process within the packaging technology.

<sup>1</sup> Krabe, D., Scheel, W., Optical Interconnects by Hot Embossing for Module and PCB Technology – The EOCB Approach. Proceedings of the 49<sup>th</sup> ECTC, San Diego, CA, 1999.

## Fatigue testing of thin metal films on substrates

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Fatigue is one of the most severe failure mechanisms of macroscopic metallic components in many technological applications such as trains or aircraft. At present, however, the role of fatigue for the reliability of micro systems has not yet been explored in detail. This is probably due to the fact that most micro-electro-mechanical systems (MEMS) are based on Si in which fatigue is not expected to be a problem. However, the introduction of metallic components in such systems can make them vulnerable to fatigue failure.

We present two methods which we have developed to study fatigue effects in thin metal films on substrates: For the first technique, thin films with typical thicknesses ranging from 0.3 to 1.5  $\mu\text{m}$  are sputter-deposited onto micromachined  $\text{SiO}_2$  cantilever beams. The beams are then cyclically deflected with a frequency of typically 45 Hz using a nanoindentation system. The second technique involves the deposition of thin films onto an elastic polyimide substrate. This film/substrate composite is then fatigue tested under tension. During the test, the elastic-plastic film undergoes asymmetric tension-compression cycles. Cu and Ag films were tested by one or both techniques, lifetimes recorded, and the damage morphology investigated. It was found that the damage extended over the entire film leading to severe degradation of its electrical and mechanical properties. Specifically, a detailed microscopic examination, including focused ion beam and transmission electron microscopy, revealed that inside large grains extrusions were formed while in regions with fine grains intergranular cracks occurred. Underneath the extrusions, large voids were observed at the interface between film and substrate. Lifetimes of the films were determined as a function of strain amplitude and film thickness. As expected, lifetime decreases with increasing strain amplitude, however, no significant effect of the film thickness on the lifetime was found. The ultimate aim of our studies is to obtain a fundamental understanding of fatigue mechanisms in small volumes which is required for a thorough reliability assessment of metallic microsystems.



## Surface modification due to technological treatment evaluated by SPM and XPS techniques

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Thin film organic polymer like Photo BCB Cyclotene™ (Dow Chemical Company) is a well suited dielectric material for the standard thin film process at the TUB/Fraunhofer IZM. The advantages of BCB are the low dielectric constant and dielectric losses, minimal moisture uptake during and after processing and very good planarization. Therefore BCB can be used for many applications in electronic packaging. Since 1994 the advantage of the photo-BCB technology has been demonstrated at the TUB/FhG-IZM for MCM-D and CSP.

Surface modification of Photo BCB due to dry and wet etching techniques is a well known procedure to increase surface quality and layer adhesion. Reactive Ion Etching (RIE) is one of the suitable methods for surface topography modification and via cleaning in thin film technology. To achieve best results in surface quality like roughness, adhesion, planarity and purity RIE parameters have to be optimized and surface topography has to be evaluated.

Because of the high planarity of BCB surfaces some problems in surface topography characterization using Scanning Electron Microscopy (SEM) occur. SEM investigations after RIE treatment with high content of fluorine gas and at low etching times become more and more difficult because of the very low roughness (on the order of tens to hundreds of angstroms). The result are images with a high noise level.

The influence of chemical reactions between the reactive plasma and the BCB surface couldn't be evaluated with common Energy Dispersive X-Ray Spectroscopy (EDX) because of the very low concentration of surface contaminants and their very low layer thickness.

To overcome these problems in surface characterization after surface modifications in topography and roughness due to plasma treatment measurements with higher lateral and vertical resolution become necessary. That's why influences of RIE parameter variations like gas composition and etching time to surface topography and roughness were investigated using Atomic Force Microscopy (AFM).

Surface contamination and effects of chemisorption due to RIE treatment were inspected by X-Ray-Photoelectron Spectroscopy (XPS).

The presented results indicate that the plasma process in thin film technology using photo BCB has a strong influence to surface morphology and roughness of the dielectric layer. The knowledge of optimal parameters like etching time and content of the

fluorine gas allow to enhance surface purity, via cleaning and layer roughness. AFM measurements have shown that surface roughness decreases at a given etching time changing the content of fluorin gas from a low content to a higher one. Otherwise the surface roughness of spin coated BCB increases with increasing etching time at a given gas composition. Optimization of both parameters can lead to a reduction of process time and improvement of the surface quality.

First time at all the influence of the so called short descum to surface topography and roughness could be shown performing non contact AFM and tapping mode AFM measurements. High surface roughness values and high content of fluorin gas lead to chemical surface modification due to interlocking of gas components of the plasma treated surface. Such chemical modifications were detected using XPS.

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## Modeling piezoelectric composites for adaptronics

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New piezoelectric thin fibers offer the possibility to make composites which have both good actuatoric and sensoric properties as well as high flexibility with respect to mechanical adaptation. The paper considers micromechanical models relating the homogenized (effective) properties of such composites to the properties of the constituents. Besides effective mechanical, electrical, piezoelectric and pyroelectric properties this includes also passive damping properties by electrically shunting of the material, and the poling process, which may be disturbed by the fiber-matrix interaction. In addition to the pure piezoelectric fibers, hybrid fibers consisting of a strong core fiber clad by piezoelectric PZT material can be considered.

The model mainly rests on the analytical evaluation of the relevant field equations making use of the electroelastic Eshelby tensor. Thus not only regular but also composite microstructures with a certain degree of randomness can be evaluated. Special attention is given to the analysis of local field intensifications which may cause mechanical or electrical damage inside the composites. Based on such results, life time assessments should become possible.

In combination with experimental data the model can be used to assess how effective the piezoelectric properties are utilized and it may be helpful in the design of optimized composite materials.

## Synthesis, dielectrical and dynamic mechanical characterisation of new thermoplastic polyurethanes for pyro- and piezoelectric applications

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In recent years, polyurethanes were suggested and studied as candidates for pyro- and piezoelectrical applications. They are thermally more stable and easier to process than the often used polyvinylidene fluoride. In these materials, a field-induced dipole orientation can be stabilised or a spontaneous dipole polarisation can be generated through the formation of hydrogen bonds between segments of different chains, like it was found for odd Nylons.

Thermoplastic polyurethanes (TPU) consist of soft and hard segments. Due to the formation of strong hydrogen bonds between the urethane groups, the polar hard segments are thermally stable up to 180 °C depending on their segment lengths. In addition, these materials show a glass transition within the unpolar soft segments. The glass-transition temperature depends on the hard-segment content. It will strongly influence the stability of the electric polarisation.

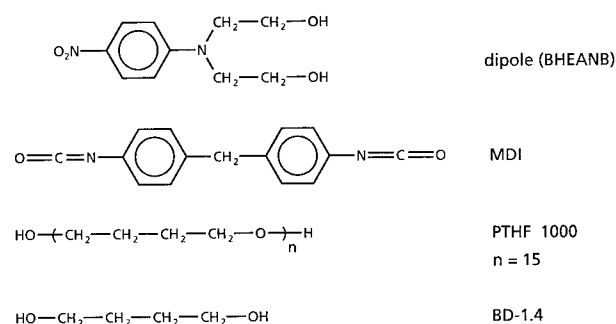


Figure 1: Chemical structures of the polyurethane components

Here, molecular dipoles are incorporated into the hard segments in order to increase their polarisation. Furthermore, the ratio between the soft phase and the dipole-containing hard phase is varied in order to enhance the thermal stability of the material. Dielectric spectroscopy and dynamic mechanical analysis are employed to study the influence of these structural variations.

Polyoxytetramethylene (PTHF1000) (Fig. 1) is normally used as the soft segment. By increasing polyoxymethylene chain length (PTHF2000 and 4000) the phase separation between hard-segment and soft-segment domains can be enhanced. The hard segment is composed of the diisocyanate component, 4,4'-methylene bis(phenylisocyanate) (MDI) and the chain extender butane-1,4-diol (BD14), as well as the covalently at-

tached molecular dipole bis-(2-hydroxyethyl)amino-4-nitrobenzene (BHEANB). The dipole moment of BHEANB is 8.5 D as calculated with the AM1 module of the Spartan program package. The polyurethanes were obtained in dimethylformamide (DMF) solution. Free-standing films with thicknesses ranging from 25 to 55  $\mu\text{m}$  were prepared from the solution by coating with a doctor blade onto glass substrates. The substance is thermally stable up to 200  $^{\circ}\text{C}$  as determined with thermo-gravimetric analysis. The relaxation behaviour is studied by means of dynamic mechanical analysis (DMA) at a frequency of 1 Hz as well as frequency-domain dielectric spectroscopy from 20 Hz to 1 MHz, both in a temperature range from -150  $^{\circ}\text{C}$  to +170  $^{\circ}\text{C}$ .

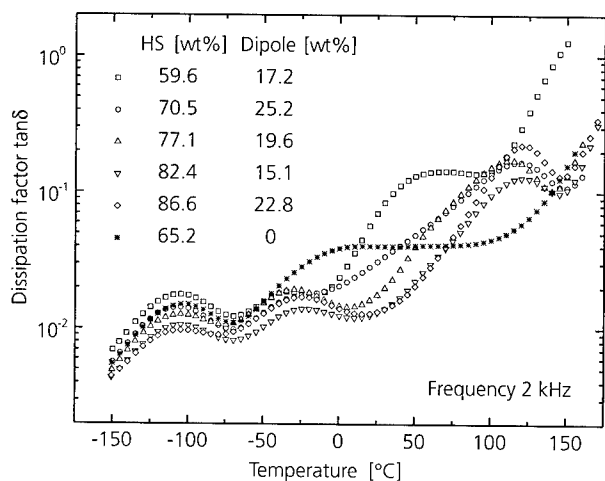


Figure 2: Dissipation factor at 2 kHz versus temperature for five TPUs with different hard-segment and dipole (BHEANB) contents as indicated

Starting with the PTHF 1000, free-standing films with a hard-segment content between 59.6 and 86.6 wt% were prepared. In this range, dipole contents between 15.1 and 25.2 wt% have been achieved, depending on the molar ratio between the hard-segment components. The glass-transition temperature as determined with differential scanning calorimetry increases with increasing hard-segment content from 40.7 to 83.5  $^{\circ}\text{C}$ . Three relaxation regions are found in the dipole containing polymers, designated as low-, intermediate- and high-temperature

relaxations (Fig. 2). The latter is not found in the spectrum of the reference polymer without any dye molecules, which indicates that the high-temperature relaxation is caused by the BHEANB dipoles.

The intensity of the low-temperature relaxation decreases with increasing hard-segment content. Therefore, it is attributed to local molecular motions within the soft segments. The other relaxations show more complex behaviour, depending on both, dipole content and hard-segment content. With increasing hard-segment content, the high-temperature relaxation shifts to higher temperatures indicating better thermal stability of the polarisation from the BHEANB dipoles. However, the relaxation strength does not necessarily increase with increasing dipole content. DMA shows similar results, particularly in the high-temperature relaxation region, indicating a close relation between dielectric relaxation and viscoelastic properties.

The PTHF 1000-based material proves to be only partly phase separated. Its pyroelectric activity is relatively weak and can be explained by dipole-density changes upon thermal expansion and contraction. Higher and application-relevant pyro- and piezoelectric coefficients are expected for more phase-separated structures with a non-vanishing net dipole moment of the hard segments.

### **Acknowledgement**

Financial support from the Stiftung Industrieforschung (Project U43/97) is gratefully acknowledged. The authors are indebted to Joachim Müller for carrying out the DMA measurements, to Reinhard Hentschke (MPI Mainz) for calculating the dipole moment of BHEANB and to Harald Goering for helpful discussion concerning the DMA-results.

## Optical properties and film stress behavior of Silver reflection coatings for micro mirrors

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High reflecting mirrors are needed for any kind of power laser applications. Micro mirrors for laser scanning applications are developed in the Center of Microtechnologies at the Technical University of Chemnitz [1, 2]. The mirrors have surface areas of some square millimeters and a thickness between 5 and 50  $\mu\text{m}$ . The material of the mirrors is crystalline silicon. Because of the low mirror thickness the stress of mirror coatings is as important as the reflectance (the bowing caused by film stress is inverse proportional to the square of the substrate thickness).

High reflectances at three wavelengths are needed for color laser display applications. This demand can be satisfied either by dielectric multilayer coatings or by metallic films. Dielectric multilayer coating design is more complex for three wavelengths than for a single one, technological requirements are very high and for a better than a metallic reflection the film stack becomes considerably thick. Therefore, a high reflecting metallic mirror coating may be a more practicable solution for microsystems.

Silver has the best reflectance of all metals in the visible spectral range including the red, green and blue spectral color. Furthermore, the low light polarization of silver guarantees a high reflection even for p-polarised light at large angles of incidence. A drawback of silver as material for reflection coatings is the chemical instability of the unprotected surface leading to a fast reflection decrease in environmental air. Furthermore, the thermal instability of the silver oxide results in problems during the deposition of oxide protection films which are the widespread optical materials.

In this paper silver based surface reflection coatings are presented. The micro mirrors consist of silicon as substrate, Titanium as sticking layer, Silver and one or two protection layers. Transparent fluoride or nitride films have been found as practicable for the silver protection. For each of these groups an example has been investigated: a CVD-CF-polymer film and an  $\text{AlSi}_{1\%}\text{N}$ -film. For the polymer films only an ex situ preparation was possible. The polymer protected silver mirror has a similar reflection like pure silver which is due too low absorption values, a sharp interface and the low refraction index. Moreover the film has good protection properties. However, the thermal stability of the available polymer films was too low. Since for the micro mirror device technology a stability up to at least 350  $^{\circ}\text{C}$  is required, the thermal stable  $\text{AlSi}_{1\%}\text{N}$  film has been examined in detail. This film is in situ deposited on the silver by R.F. sputtering of an  $\text{AlSi}_{1\%}$  target. The reflectance of the mirror films was precisely measured. The film reflectance was higher than calculated using standard literature values of the optical constants of silver [3]. The stability has been proved using sulphur containing environmental tests.

For a perfect long term stability, the protection coating has to be a defect free barrier layer for sulphur and oxygen. To enhance the safety of protection,  $\text{TiO}_2$  has been used

as second protection film on top of the  $\text{AlSi}_{1\%}\text{N}$ .

Film stresses are important properties especially for micromechanical applications. Therefore, these properties depending on deposition parameters have been examined for the Ti, Ag, AlSiN and  $\text{TiO}_2$ -films. The results will be presented and discussed.

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Academic Press San Diego - London - Boston - New York - Sydney - Toronto



## Stress-induced lateral deflections of microstructures

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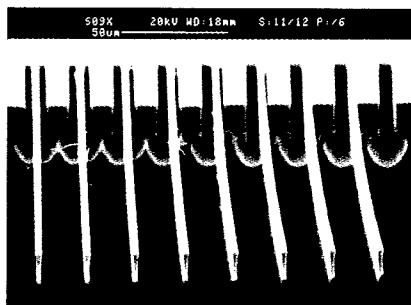
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Microelectromechanical systems (MEMS) based on single-crystalline silicon have a wide variety of applications because of the well-known excellent mechanical properties. Single mask high-aspect ratio micromachining [1] offers principal possibility of monolithic integration with electronics on a cost-effective base. However, for several applications, e.g. differential capacitor accelerometers, trenches with differing widths are required. Depending on the step coverage of the used deposition methods (PE-CVD, sputtering) the varying trench width surrounding the beam results in asymmetrical sidewall deposition onto the beam causing its lateral deflection. Recent investigations [2] have shown the principal dependence of beam deflection as a function of the asymmetrical silicon oxide layer thickness deposited by PE-CVD. Since the investigated silicon oxide films show compressive stress the beams were bent towards the narrow trench. Therefore the maximum lateral deflection is limited by the narrow trench. However, for use in potential applications (e.g. as an actuating structure) a large possible displacement is desirable. Consequently, the direction of deflection has to be changed to the opposite

direction. This can be achieved by using films showing tensile stress.

In this paper we report on investigations extended to films of silicon nitride and silicon oxynitride deposited by PE-CVD and silicon oxide grown thermally. Long and narrow beams suspended either from one end or from two ends were analyzed. Improved geometrical description of cross section of the beams was used for FEM-simulation to support experimental results.



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## Finite element techniques for flaw analyses in piezoelectric micro-components

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Piezoelectric, ferroelectric and dielectric ceramics or polymers are widely applied in Micro Electro Mechanical Systems (MEMS) to supply the essential sensing and/or actuating functionality. As a consequence of their integration into MEMS, these smart materials may be exposed to extraordinary high mechanical and /or electrical static, dynamic or cyclic loading. Therefore, problems of fracture and fatigue play an important role for the optimum design and the reliable service performance of MEMS. Fracture mechanics analyses and safety concepts have to be applied to crack-like defects in piezoelectric bulk materials or in interfaces structures.

The authors have developed efficient numerical methods for solving the singular coupled electrostatic and anisotropic elastic boundary value problem for 2D cracks in homogeneous piezoelectric structures of arbitrary geometry under electromechanical loads and for determining the fracture controlling parameters [1]. In the present paper, the analytical and numerical techniques are extended to heterogeneous structures composed from piezoelectrics, metals and/or silicon (typical for MEMS). Special attention is paid to cracks, edges and notches at interfaces and to non-symmetric mixed-mode loading of the cracks.

A semi-analytical method has been elaborated to find the exact asymptotic solutions for the electromechanical crack tip fields at the above mentioned crack configurations. Based on these asymptotic expansions, special finite element techniques are developed for analysing complex micro-structures:

1. In the first method, the complete asymptotic solution is embedded into a special region surrounding the crack/notch tip, which is coupled with the regular finite elements. This technique delivers directly the coefficients of the asymptotic expansion, i. e. the stress intensity factors for notches, cracks and interface-configurations.
2. For cracks, the required type of singularity can be simply achieved by modifying standard finite elements by means of the so-called quarter-point distortion. This technique gives an easy way to compute stress intensity factors for mixed-mode crack configurations.
3. The modified crack closure integral technique was numerically implemented in order to compute the electromechanical energy release rate for arbitrary 2D cracks and interface cracks.

The efficiency and the performance of all the suggested techniques are verified with sample problems and compared to each other.

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## New investigations in materials research by confocal laser scanning microscopy

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Modern Confocal Laser Scanning Microscopy (CLSM) provides powerful solutions for numerous problems in materials research, failure analysis and quality assurance. The major industrial applications range from micro system techniques, polymer and ceramics industry to food industry, whereas in the scientific area metals research, corrosion research, crack analysis and forensics indicate only a few of the numerous possible applications.

The basic principle of a CLSM is the following:

A laser beam is directed through an optical microscope and reaches a sample as a diffraction limited spot. Light (reflected, scattered or emitted) is captured by microscope objective and reaches a detector via a confocal pinhole. This pinhole is placed in such a way that only light from the objective lens focal plane can pass through the pinhole. Out-of-focus contributions are blocked. This depth selection mechanism of the confocal LSM is the prerequisite of creating thin optical sections of the sample. By moving the laser beam point by point over the sample, together with a precise focusing mechanism, three-dimensional image stacks can be acquired.

Imaging methods, such as, reflected light and fluorescence, can provide new information about the sample material. Other scanning techniques such as electron and interference microscopy cannot provide this information in its entirety:

- Non-contact, three-dimensional representations of structures can be recorded without time consuming sample preparation
- The volume inside of semitransparent objects can be imaged non-destructively up to a depth of a few hundred microns
- Multiple profiles can be extracted from these 3D images or can be recorded directly along a free spline track to follow for instance a crack pattern to be scanned
- Quantitative data about distances, heights, angles, areas and volumes, all given directly in mm, mm<sup>2</sup> or mm<sup>3</sup>, degrees or percentages, are available
- Statistical values like micro roughness parameters (2D and 3D) can be evaluated
- Fluorescence images can provide spectroscopic information that can be correlated to local variations in the chemical and physical properties of the sample
- Time based changes of surfaces or volumes can be recorded from a few seconds to several hours where data acquisition can be triggered by external events
- Completely automated solutions can be implemented.

The Confocal Laser Scanning Microscope is a highly beneficial complement to classical light microscopy or to scanning electron microscopy (SEM) and it has become the method of choice for those applications that require a fast, direct and accurate quantitative analysis of 3D microstructures. With a high degree of motorization and the use of comfortable 3D display and processing software packages, the modern day Confocal Laser Scanning Microscope has opened the way for new and exciting research as well as routine applications in industry.

## Temperature influence on current suppressing effect in SiC Schottky diode

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By manufacturing of large area Schottky contacts to semiconductor surfaces, the interface area will have a number of inhomogeneities. These inhomogeneities lead to the local characteristics at the interface, which may lead to current suppressing effect. The electrical and thermal behaviour of such devices is studied using SPICE simulation package SCHOTSIC, developed at the Department of Electronics, TTU. This package simulates both electrical and thermal processes in device using standard SPICE environment. Iterative simulation process is used to couple with self-heating effect (see e.g. [1]).

Simulations with different ambient temperatures (300K-900K) have been finished. The calculations show that the current suppressing effect depends strongly on temperature.

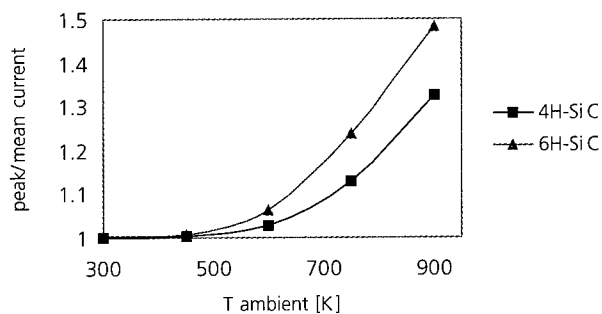


Figure 1: Peak current dependence on ambient temperature

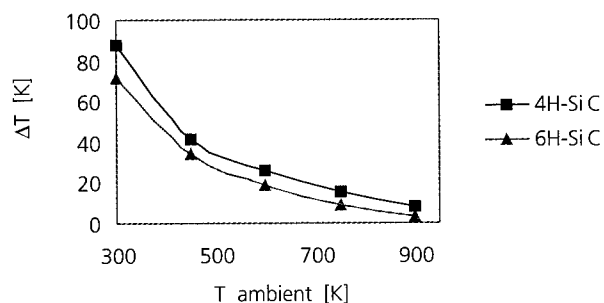


Figure 2: The self-heating effect dependence on ambient temperature

Figure 1 shows the peak current dependence on applied temperature for 4H-SiC and 6H-SiC large area Schottky structure. This relatively large temperature dependence is mainly explainable with the change of the resistivity of semiconductor material under high ambient temperature. The problem is especially important in region of high temperature. Higher peak currents result in relatively higher local temperatures. Unfortunately, this quality of mentioned behavior leads to danger to destroy the structure through hot spot. The peak is higher in case of 6H-SiC semiconductor because of its strong unisotropic characteristic of the crystal.

It is also important to mention that ambient tempera-

ture is not the same as temperature of the device. The self-heating effect is higher on lower temperatures (Figure 2.). Higher semiconductor intrinsic resistance in case of lower temperatures explains this fact. Although the self-heating effect is low on high temperatures, the effect becomes important on low temperatures because of thermal expansion.

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## Deformation behavior of micromirror arrays under optical power

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Conceivable applications of micromirror arrays are image projection, laser machining and mask-less lithography among others which require transmitting a laser beam with an optical power of several Watt. The influence of the partially absorbed power on the surface deformation by the caused temperature rise is discussed in this contribution on the example of bulk-micromachined electrostatically driven micromirror arrays.

Analytical approximations of the thermal resistance have been applied to predict the thermal resistance by conduction in the torsion beams and in the electrode gap. The influence of the thermal resistance of the mirror frame in series with the torsion beams and the effect of the carrier depends on the complete set-up of the deflection unit and has been neglected. Furthermore, a thermal CFD analysis using ANSYS/FLOTRAN has been carried out.

In order to experimentally find the thermal resistance while the thermal capacitance is given by materials and mirror size, single micromirrors have been mediate heated by Joule loss. The decay coefficient has been fitted on base of the temperature abduction curve after switching off the heating. The experimental results reflect, that the thermal resistance and maximum power dissipation are well predictable by the applied models.

To test the deformation of the mirror at high temperature and determine the maximal allowable mirror temperature, the surface topology is measured by a phase shift interferometer while the array is placed in a heating chamber.

One can conclude that heat conduction in the gap between the mirror plate and the glass is the major quantity for thermal energy transfer. The dominance of conduction in the gap rises when diminishing the gap size. Assuming less than 10% absorption loss of incident light power at the mirrors we expect a maximum deflected light power of more than 30 Watt in case of cw-lasers.



## Porous silicon as new micro material for actuated 3D flip-up structures

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This paper reports a new surface micromachining method based on porous silicon formation followed by electropolishing. The electrochemical etch process generates in a single step a plate of porous silicon, releases it from the substrate without using a sacrificial layer and lifts it out of plane. The plate can be tilted by a thermal bimorph actuator. Its application can be seen in microoptical systems like scanner mirrors or tunable optical filters or in microfluidical systems as valve.

The fabrication process uses two photolithography levels on ordinary silicon  $p^+$  substrates. The lower layer is a mask of structured LPCVD silicon nitride ( $\text{Si}_3\text{N}_4$ ) to define the regions on the silicon substrate to be porosified. The second layer on top consists of evaporated Cr / Au tracks to define the heater resistor for the thermal bimorph actuator with contact pads. This wafer is electrochemically etched in a HF / ethanol solution. First, a current of about  $100 \text{ mA} / \text{cm}^2$  is used to porosify a layer of silicon of the desired thickness to form the plate where the  $\text{Si}_3\text{N}_4$  mask is open. After that the source switches to a higher current that leads to isotropical electropolishing under the plate and releases it from the substrate (Fig. 1). The porous silicon already built will not be affected by the ongoing processing. The plate is held by two flexible actuator arms at the sides. The silicon nitride mask is not fully open in the actuator region but perforated to reduce the etch current locally. This makes the arms thin and flexible and keeps them connected to the substrate. An inhomogeneity in the porous layer of the arms caused by the increasing etch interface area during the process leads to mechani-

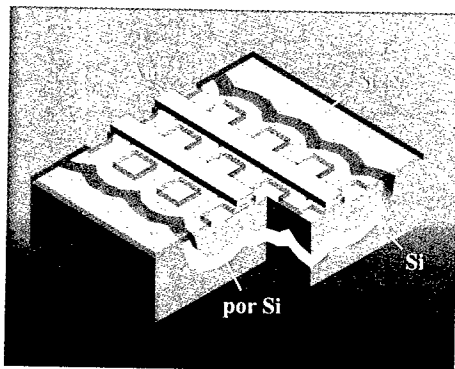


Figure 1:  
Actuator arm made of porous silicon,  $\text{Si}_3\text{N}_4$  and Au, released from the substrate by electropolishing. Removing the  $\text{Si}_3\text{N}_4$  mask will make the structure lift up.

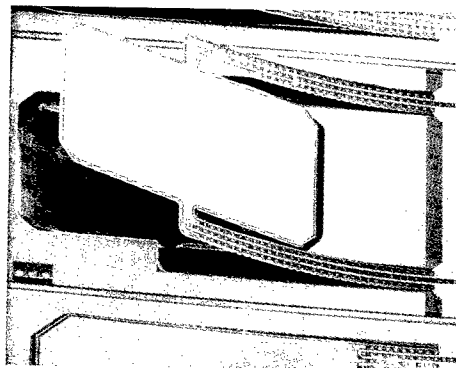


Figure 2:  
SEM picture of a free-standing, mobile, flat plate of porous silicon lifted out of plane and suspended by two actuator arms.  
Dimensions: 1850mm x 1100mm x 30mm

cal stress that lifts up the structure. Finally the wafer is left in the HF / ethanol solution to dissolve the silicon nitride mask. The whole etching process takes less than one hour. Large, flat, flip-up plates of porous silicon have been realized with a typical thickness of 30 nm and areas ranging from 250  $\mu\text{m}$  by 750  $\mu\text{m}$  to 2400  $\mu\text{m}$  by 4000  $\mu\text{m}$  (Fig. 2). In the rest position they are lifted by two arms 10° to 90° out of plane. They can be thermally actuated by applying up to 5 Volts. The filter plate can be set into mechanical resonance. Depending on geometry, the fundamental frequency is between 100 and 3000 Hz. The angle of deflection can be as large as 30° at resonance. The device can be oxidized to eliminate absorption of visible light and induce additional stress, which bends the actuator into a higher rest position. This new surface micromachining technology for creating free-standing structures using a minimum of process steps offers new possibilities in micromechanics and microoptics.

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## Light emitting devices based on Al-porous silicon Schottky junctions on the sapphire substrates for microdisplay applications

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The fabrication technology of light emitting devices based on Al-porous silicon (PS) Schottky junction on the transparent sapphire substrate has been developed. White light emission, visible by naked eye, is observed under reverse bias.

The current level at which the EL starts is around 1 mA for devices of  $2.3 \times 10^{-3} \text{ cm}^2$  area. The light emission intensity increases with increasing of current density. EL spectra were broad, covering the whole visible range. The pixel size could be less than  $2 \text{ }\mu\text{m}$ . The time stability was excellent for all tested devices: the EL intensity did not show remarkable changes, even after more than one month of continuous light emission at currents lower than thermal breakdown.

## Lead-free soldering – are you ready for this global trend?

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Lead-free soldering for electronic industry is a segment of global trend toward lead-free environment. Although initiated in U.S. in early 1990's, it advanced much more rapidly in Japan and Europe. This differentiation in Pb-free progress triggered great concerns of users of Pb-containing solders about maintaining business opportunity, therefore further expedites the advancement of Pb-free soldering programs. The favored Pb-free solder alternatives vary from region to region. However, in general, high tin alloys are preferred, including Sn/Ag, Sn/Cu, Sn/Ag/Cu, Sn/Ag/Bi, and various versions of those alloys with small amount of additions of other elements, such as Sb. Sn/Ag/Bi systems are used in some Japanese products already. However, Sn/Ag/Cu systems are more tolerant toward Pb contamination than Bi-containing systems, therefore are more compatible with existing infrastructure for the transition stage. Pb-free surface finishes for PCBs include OSP, immersion Ag, immersion Au/electroless Ni, HASL Sn/Cu, Sn/Bi, electroless Pd/electroless Ni, electroless Pd/Cu, and Sn. The challenge for components is greater than for solder materials or PCBs. Although some Pb-free surface finishes for components exist, such as Sn, Pd/Ni, Au, Ag, Ni/Pd, Ni/Au, Ag/Pt, Ag/Pd, Pt/Pd/Ag, Ni/Au/Cu, Pd, and Ni, the performance remains to be verified. In addition, options for higher melting temperature solder is still not available for high temperature applications, including first level interconnect within the components. Thermal damage can be a concern for both PCBs and components.

## Mechanical and thermomechanical properties of Al-SiC-MMC

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With increasing concentration of energy in microelectronic components heat dissipation becomes a decisive design criterion, in particular for high power electronics. E.g. in power modules the metalized ceramic substrates are soldered on one side to the Si based chips and on the other side to a base plate. The changes in ambient temperature and moreover the heating during operation causes thermally induced stresses between the different materials because of their different coefficients of thermal expansion. Damage of the interface layer is enhanced by temperature cycles causing thermal fatigue of the solder joint due to crack propagation, which reduces the heat transfer and thus the electric efficiency and service life time of the modules. Thermally matching, high thermal conductive material can be achieved e.g. by Al matrix composites containing high volume fractions of SiC particulates. The low thermal expansion of the ceramic constituent reduces that of the Al matrix according to its volume fraction within the composite. The reliability of the sandwich module will be increased essentially by approaching the thermal expansion of the substrate.

Ceramic preforms of more than 40 vol.% SiC particulates can be prepared and then infiltrated by the molten matrix. Differently processed Al-SiC compound have been investigated. The local distribution and volume fraction of the SiC particulates was investigated by light microscopy on polished cross sections in two orthogonal directions. Quantitative image analysis was applied to determine the size distribution of the particulates. The coefficient of linear thermal expansion was determined by dilatometric measurements in the temperature range between room temperature and 300 °C. The heat capacity was measured in the temperature range of -55 °C to +200 °C by differential micro-calorimetry. The mechanical properties were determined by four point bend tests at room temperature. Elastic modulus, flexural strength, elongation at rupture and yield strength have been deduced from the force - strain curve.

The metallographic investigations revealed that the samples contain different size classes of SiC particulates. Particle size ranges, volume fractions and particles orientation were obtained from the quantitative image analysis. The volume fractions of SiC correlate to the CTE which decreases with increasing content of particle, whereas thermal conductivity and Young's moduli increase. The flexural strength increases with the ductility of the material. The heat capacity is the same within the experimental accuracy for all materials tested.

**Keywords:** SiC-particulate reinforced Al, Al-matrix composite, heat sink, insulated gate bipolar transistor, thermal expansion matching, mechanical properties, micro-structure.

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## Study of thermal deformation in BGA assemblies using FEM<sup>1</sup>

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In this paper, FEM method is used to investigate the deformation behavior of BGA assemblies, including PBGA and CBGA, caused by thermal cycle. In the FEM model, the BGA assembly is considered as three layers of material: the BGA module, the solder ball, and the printed circuit board (PCB). The material of each layer has different property parameters. As we all know, numeric simulation using FEM method requires sophisticated computer equipment and expensive software packages. In order to cut short the calculation time and expenditure, the calculation procedure is separated into two steps. In the first step, a simplified model, in which the solder ball layer is considered as solder poles, is used to calculate the total deformation of the assembly. After

we get the total deformation, the maximum deformation site on the assembly can be found. And in the second step, for the maximum deformation sites, finer meshes were used to calculate the detailed stress-strain distribution field of the specific solder joint. The stress-strain distribution field is the key factor for the analysis of the reliability of BGA assemblies.

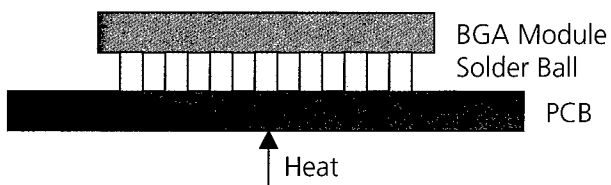


Figure 1: The simplified model used to calculate the total deformation

ANSYS software package is used to carry out the FEM calculation. The assumption is that the heat is applied to the specimen from the center of the BGA assembly (Fig 1), and the size of the PCB is considered as ten times larger than the BGA module, the maximum temperature in the center of the BGA module is 100 Degree Celsius. Calculation result shows that, for the PBGA assemblies, with the increase of the temperature, the deformation change from ball-shape out-of-bending to saddle-shape warpage, and with the decreasing of the temperature, the deformation changes back to ball-shape. But for the CBGA assemblies, the deformation is always ball-shape out-of-bending. This is caused by the great variation of Yang's Module between the plastic and alumina ceramic. The maximum deformation site of the assemblies is found at the fringe of the BGA module.

<sup>1</sup> NSFC General Project, Grant No.: 69776029.

Non-destructive testing methods based on the Real-time Holographic Interferometry and Moiré Interferometry are used to measure the out-of-plane and in-plane displacement deformation. The testing result is coincided with the FEM calculation.

**Keywords:** BGA (Ball Grid Array), FEM, Thermal Deformation, Real-time Holographic Interferometry, Moiré Interferometry

## Lead-free solders for flip-chip-technology

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Engineering of materials means a synergistic development of both materials and processes.

The development of lead-free solders for flipchip-technology is presented as an example for the complex relations between material properties and deposition processes. Eutectic tin-silver and eutectic tin-zinc are discussed with respect to the plating processes used for the deposition of the materials.

Bump plating is discussed for waferbumping and for bumping of PCBs.



## Design optimization for electroplated micro-springs

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### Introduction

Multi-Chip-Module technology requires the information about the proper function of every single sub-chip. Several solutions for this system inherent problem were presented. One promising approach is the Composite IC concept introduced in the 1994 National Roadmap for Semiconductors. According to this concept a Composite IC is an integrated circuit assembled from sub-chips mounted on a large back-plane chip. Surface micromachined micro-springs are used to provide reliable interconnections to the backplane chip. The first assembly step is to connect the untested die temporarily to the micro-springs. The complete system can then be tested. Defective components can be replaced and the test can be repeated until the whole system meets its specifications. In a second step the chips are rigidly fixed through a reflow process [1][2]. Suitable micro-springs were presented for the first time in [3]. This paper deals with the optimization of this spring design.

### Design and fabrication

Fig. 1 shows a scalable interlaced micro-spring design which fulfills the mechanical requirements of the Composite IC concept. This design allows a fine pitch, high vertical travel and sufficient contact force in order to form reliable electrical contacts. The micro-springs are fabricated in a nickel electroplating process [4] with an evaporated gold layer on top to achieve low contact resistance. The current design has a pitch of  $650\text{ }\mu\text{m}$

and the thickness varies between  $8\text{ }\mu\text{m}$  and  $20\text{ }\mu\text{m}$ . The next step is to shrink the design suitable for Flip-Chip technology. The goal is to provide a vertical travel of  $50\text{ }\mu\text{m}$  at a load of  $20\text{ mN}$  and a minimum thickness. Downscaled micro-springs with a pitch of  $130\text{ }\mu\text{m}$  are currently under fabrication.

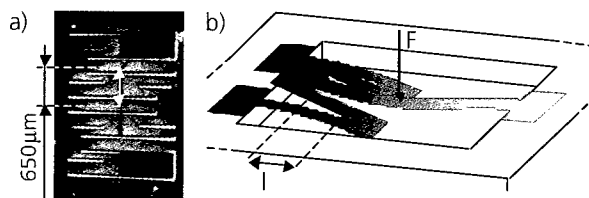


Figure 1: a) Three interlaced electroplated micro-springs  
b) Deflected model of a single micro-spring

### Measurement and simulation

In experiments force was applied to the micro-springs. Fig. 2 a) shows the relationship of applied force with respect to the deflection at the contact point shown in fig. 1 b).

To predict the spring characteristics a finite element model was developed using the FEM-tool ANSYS. To minimize the numeric effort for calculating deflections a 3D shell model with rotatoric and translatoric degrees of freedom was utilized. Pure elastic behavior was assumed and nonlinear analyses of the deflections were performed. Force vs deflection measurements and simulations show a good correlation at different thicknesses with an estimated Young's modulus of 80 GPa (fig. 2 a)). The thickness has a strong influence on the stiffness of the micro-spring. Simulated force vs deflection

curves for the shrunk design with a pitch of 130  $\mu\text{m}$  are shown in fig. 2 b). At smaller deflections a higher contact force can be achieved. Fig. 2 b) shows that the goal of 20 mN at a deflection of 50  $\mu\text{m}$  can be performed with the downscaled design.

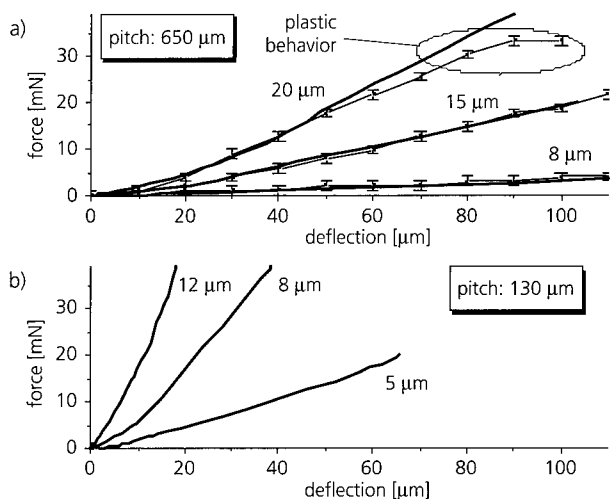


Figure 2:  
Force versus deflection for different thicknesses  
a) 650  $\mu\text{m}$  pitch, measurement (scatter) and simulation (line)  
b) 130  $\mu\text{m}$  pitch design, simulation

In the paper design optimization of the micro-springs will be presented. Attention is paid particularly to an increased spring constant, e.g. through a reduction of the thigh length  $l$  (fig. 1 b)), and to prevent stress concentrations in the structure, e.g. by smoothening the corners. For exact determination of the occurring bulk stress and strain a finite-element model with volume elements was developed, the results will be discussed in the paper.

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## Carbon components for micro-world machines

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Micro- and nano-sized carbon structures have been synthesized and isolated. Carbon nano-pipes have been synthesized in hydrothermal fluids with sub-micron outside diameters and inside diameter to wall thickness ratios of about 5:1. These nano-pipes occasionally closed and contain fluid inclusion visible under the high vacuum of TEM microscopy demonstrating that the pipes are very leak-tight. Lengths of nano-pipes up to 20 mm have been obtained which are open from end to end. High flexibility is expected of these nano-pipes. The nano-pipes are easily cut with a focused laser and air oxidation.

Carbon needles having a stepped longitudinal dimension starting at 500 nm diameter and terminating in 10 nm x 1  $\mu$ m long nano-needles have been found. These novel graphitic structures show a high degree of crystalline perfection and by virtue of a very high base to tip diameter ratio, render them capable of manipulation. These carbon structures show very good conductivity by virtue of the capability to easily image such fine and long nano-structures in an FESEM microscope. The potential of the produced carbon nanostructures as fluidic and electrical components has been considered.

## Development of micro scanning devices actuated with PZT thin films

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$\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$  thin film is widely used in micro-electro mechanical systems recently because of large piezoelectric and pyroelectric properties. Several deposition processes have been developed to prepare PZT films such as sol-gel and sputtering processes. Due to low deposition rate and high residual thermal stress in these two methods, the pulsed laser ablation process, which has high deposition rate (3 $\mu\text{m/hr}$ ) at room temperature, has become a promising technology for MEMS applications. This process technology is carried out in an ultra high vacuum chamber by pulsed KrF excimer laser ablation with radiation of FAB treatment.

The design, fabrication, and characterization of micromachined scanning beams and mirrors actuated by laser ablation PZT films are investigated. The actuation principal is based on a bimorph beam structure, which consists of an oxide layer and a piezoelectric PZT layer. The scanning is achieved by applying AC voltages at resonant frequencies to actuating bimorph beams. Variant scanning beams and scanners composed of a mirror plate with actuating beams are designed.

Only three lithography steps are required for fabrication. First, all layers are deposited. (Thermal oxidation  $\text{SiO}_2$ ; Pt/Ti sputtering as the lower electrode;  $\text{Pb}(\text{Zr},\text{Ti})\text{O}_3$  prepared by laser ablation method with post annealing at 700C for 4 hours; Pt/Ti sputtering onto PZT layer as upper electrodes). Secondly, ECR and RIE etching systems are used to structure the front side. Finally, the 5 $\mu\text{m}$  thick structure is released from bulk silicon successfully by ICP deep silicon etching of the wafer backside. The structure is usually suffered from residual stress of the deposited thin films. In the present paper the double layered PZT structure was also fabricated and tested.

The scanner with a 200  $\mu\text{m}$  x 200  $\mu\text{m}$  mirror and two 50  $\mu\text{m}$  x 500  $\mu\text{m}$  actuating beams has been investigated. First the resonant frequencies of the structure were measured by using a laser interferometer and FFT-analyzer. Secondly, the vibration shape of the mirror was determined by measuring the deflections of different locations, stimulated by a function generator (16Vpp AC). Thirdly, The scanning angle was obtained by measuring the deflection of a laser beam projected onto a screen. The scanning angle of this device is quite small. A better performance could be expected in a further step by some poling or post treatments.

**Keyword:**  $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ , Thin film, Actuator, Scanning mirror, Piezoelectric, Excimer Laser Ablation

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## Non self-similar crack extension: to »KINK« or not to »KINK«?

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In conventional linear elastic fracture mechanics, a kinked branch crack is specified as an integral outgrowth of the parent crack tip, which acts as an initiator from which non self-similar crack extension can take place (Lo, 1978; Wu, 1978; Cotterell and Rice, 1980; Hayashi and Nemat-Nasser, 1981; He and Hutchinson, 1989). The physical justification for the »kink« is the presence of inherent flaws in the material body which have a small but non-zero characteristic length. The obvious attraction of such an approach is that Irwin's (1957, 1958) notion of a stress intensity factor  $K$ , which is based on self-similar crack extension, may be retained, albeit updated to the direction of the kinked branch crack.

However, such a notion implies an incompatible combination of physical and mathematical (or phenomenological) modelling. That is to say that when evaluating the rate of energy release  $G$  which determines crack extension, the mathematical standpoint adopted is that the corresponding differential  $dU/da$  would be based on the limiting condition that  $a/\lambda \rightarrow \infty$ ; in doing so, the physical notion that the kinked branch crack would have some small, albeit non-zero length of  $\lambda$ , may be satisfied. On the other hand, such a rationale would be inconsistent with the proper definition of a differential which is based on the application of Weierstrass' limit theorem (Belding and Mitchell, 1991) and requires that  $\lambda=0$  in the evaluation of  $dU/da$ . Furthermore, it is a moot point that, whereas on the one hand the kinked branch length  $\lambda$  is deemed to be small enough to satisfy the above condition of a differential, the branch tip is, on the other hand, considered to be sufficiently remote from the singularities at the knee of the branch for its near field to be outside their zone of influence.

As a consequence of the above inconsistency, it has been found that the definitions of the pure modes I and II stress intensity factors in the non self-similar direction by crack kink analysis,  $K_I(\theta)$  and  $K_{II}(\theta)$ , are subject to inaccuracy at small angles of  $\theta$  to the parent crack direction; at higher angles, the solution becomes intractable due to lack of convergence (Lo and Tamilselvan, 1999). This inconsistency has recently been redressed by the development of the *unified model* (Lo et al., 1996) and determination of corresponding stress intensity factors  $K_{I\theta}$  and  $K_{II\theta}$  by closure analysis, taking into proper consideration the limiting conditions specified by Weierstrass.

In doing away with the need for a kinked branch crack, the *unified model* has simplified the problem of non self-similar crack extension considerably while at the same time providing an exact solution of it, by adopting the near field of the parent crack as its basis. In particular, the hitherto intractable problem of mixed modes I, II and III fracture may be resolved. The proposed approach was first intimated in the introduction of the *unified model* for fracture by Lo et al. (1996), and is essentially a generalisation of the concepts adopted by the model for in-plane mode of deformation, to deal with the case of three-dimensional behaviour.

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## Etching of microstructures and modification of solid surfaces by low energy ion beams

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Ion beams have been frequently used for etching micron and submicron patterns when a high anisotropy of the etching process is required. Additionally, the beams became a convenient tool for modification of morphology, composition and structure of solid surfaces and thin films. In principle, these applications are based on the sputtering of atoms from target surfaces, ion beam promotion of surface diffusion and reactions of adsorbed particles, and on the ion beam enhanced diffusion of built-in elements into deeper layers.

Contrary to »classical« technologies (e.g. wet etching, plasma etching) the intensive beams of low energy ions are able to modify and to etch some refractory materials such as specific metals, synthetic minerals, ceramics, diamond-like thin films, etc.

In our contribution we will demonstrate the ability of low energy argon ions to produce optical grids and matrices for holographic grids with micron and submicron patterns, respectively. The grids were produced by ion beam etching of chromium film deposited on glass substrates by magnetron sputtering, the matrices were etched into silicon samples. The patterns were drawn into electron resist covering the chromium film or the silicon substrate by electron lithography. The etching of the patterns was carried out by 600 eV-broad-argon ion beam produced by the Kaufman ion source built in our group. SEM and AFM investigation of the final products confirmed the presence of sharp and well defined patterns etched into chromium films and silicon surfaces (periodicity 0.7 nm).

To show the ability of ions to modify surfaces of refractory materials, the results of ion beam bombardment of ceramic samples ( $\text{Al}_2\text{O}_3$ , hydroxylapatite) and silicon will be presented. In particular, the influence of ion beam parameters (e.g. ion energy, ion incidence angle) on surface modification, sputtering and chemical composition of these materials was studied. Significant changes in surface morphology and chemical composition were particularly found in the case of  $\text{Al}_2\text{O}_3$ . The surface of individual grains on the sample surfaces showed a glassy appearance and at oblique incidence angles revealed the formation of a texture with orientation corresponding to the direction of the ion beam projected into the sample surface. Modification of the chemical composition of sample surfaces was studied by XPS. It was found that the outermost layers of untreated alumina were completely formed by silicon atoms resulting from the segregation of  $\text{SiO}_2$ -based additives. With increasing ion beam energy the content of silicon and aluminium was decreased and increased, respectively. Additionally, due to the preferential sputtering, the concentration of oxygen was reduced from its stoichiometric

value (60%) to 7% at an ion energy of 1000 eV. The experiments performed on hydroxylapatite ceramics revealed a similar decrease of oxygen on their surfaces. The influence of ions at ion-beam-assisted deposition (IBAD) of thin films will be discussed as well.



## Dislocation nucleation during nanoindentation

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Controlled probe contact techniques can be used to measure forces and displacements on the nanometer scale. Thereby it is possible to initialize a highly localized stress fields into the sample. In this way it can be detected elastic and plastic deformation of solids under extraordinary conditions. For single crystals with defect free regions like cleavage face ( $\text{CaF}_2$ ) or electropolished surfaces (Si) the initial loading is pure elastic and is followed by a rapid onset of plastic deformation. This behaviour is known as »Pop-In-Effect«. It will be suggested that the stress of the elastic deformation approach the theoretical shear stress for nucleation of a first dislocation loop.

By the theory of elastic contact between surface and axially symmetric punch (Sneddon) the stresses can be calculated and compared with the value of theoretical shear stress for loop nucleation (Frank-loop). For several cubic materials like metals, for semiconductors and for ionic crystals the measured stresses are always in good agreement with the theoretical nucleation stresses.

Furthermore there exist strong correlations between the parameters of surface preparation and the stress levels for dislocation generation.

## Process and mechanical behavior of micro and nano materials

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Micro and nano materials are being increasingly used in industrial applications as structural components or functional devices. Mechanical reliability is one of the critical issues with respect to these materials. Most of them, in fact, are multi-layer systems. Due to the differences in the thermal expansion coefficients of the different layers and the thermal gradient produced during processing and operation, residual stress can occur, which can have an important effect on the mechanical behavior of the materials. Experimental techniques and numerical tools have been developed to effectively study the residual stresses involved and their effects on the mechanical behavior, material processing and microstructure of materials. This paper gives an overview of the different research projects being conducted in our laboratory in cooperation with other research institutions.

The first part presents the concept of surface nanocrystallization (SNC) of metallic materials. Three types of SNC processes are defined:

- Generation of nano structures by surface coating or deposition,
- Surface self-nanocrystallization
- A hybrid technique for the generation of nano structures on the surface

Different SNC mechanisms and possible SNC techniques are discussed with the emphasis on mechanically induced surface self-nanocrystallization. Several examples are provided on using this approach to obtain nanostructures on the surface of pure Fe and Cu, stainless steel, low carbon steel, etc. The surface characteristics of the materials are determined using X-ray diffraction (XRD) and transmission electron microscopy (TEM). Experimental evidence shows that after surface treatment, the initial coarse-grained structure of the surface layer was converted into ultrafine equiaxed grains (about 10nm) with random crystallographic orientations over a depth of 20  $\mu\text{m}$ . The grain refinement mechanism is discussed. The results also show that the increase in tensile properties (yield stress and UTS) of the material with the generation of nanostructure on the surface is quite significant. The prospects for structural and functional component applications are discussed.

The second part of the paper concerns the mechanical properties of thin films using the nano-indentation method. In this study, the load-displacement curve obtained by nano-indentation is used to determine the yield strength and hardening index with a finite element simulation. An application using an Al film on a Si substrate is presented. Nano-indentation is also used to study the effect of the thickness of a DLC thin film on the hardness of the film.

The last example given is the study of the in-depth residual stress distribution on packaging for electronic components using a combination of moiré interferometry and the high-precision incremental hole drilling method. A special finite element program was developed to determine the calibration coefficient of multilayer systems. The effect of the size of Si chips on the residual stress was studied.

## Thermal matching of a glass-ceramic-interface for a carbon dioxide sensor

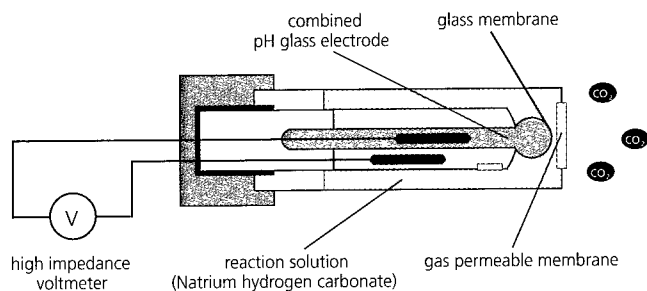
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In saturated hydrogen carbonate solutions the concentration of carbon dioxide is proportional to the pH value. This effect is used for carbon dioxide sensors of the Severinghaus type. Suitable methods for the determination of the pH value operate with pH glass electrodes, solid-state glass electrodes, metal oxide electrodes or ISFET's. But doubtless the best measurement results can be achieved only with glass electrodes. These glass electrodes have the disadvantages of a relatively high price (because of the manufacturing

ing) and of a poor mechanical stability (because of thin glass membrane). Therefore our research goal was the development of a reliable pH electrode with improved mechanical robustness using the thick film technology as well-tried mass production.

To increase the mechanical stability of the membrane



General arrangement of the carbon dioxide sensor (Severinghaus type)

of the glass electrode and to guarantee the electrolytic contact between glass and internal reference electrode we decided us for a porous carrier substrate. That carrier has meet the following demands:

- chemical inert also under the manufacturing conditions of 1000°C,
- minimum of CTE-mismatch,
- no influence on the electrical behaviour of the pH element.

We expected that ceramics could solve all requirements. Different ceramics like zirconium dioxide or titanium dioxide and different glasses were tested on their suitability. But by using pure materials the CTE-mismatch is too high, which leads to cracks in the glass layer. The best results were achieved with calcium stabilised zirconia. That's why it was necessary to adapt the CTE of the pH glass to the CTE of the zirconia. During the processing of the glass to a thick film paste, it was filled with zirconia powder. Following the effect of different shares of  $ZrO_2$  was tested.

Our paper shows the experimental procedure of the CTE-matching and gives a discussion of the results. Like many other technical problems it was because of missing material parameters impossible to compute or to simulate the reactions of different materials.

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## Control of electroplating depositions in MEMS-fabrication

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Electrodeposition processes have long been proposed for the fabrication of micro-electro-mechanical systems »MEMS«. The aim of the presented project is directed to the standardization and improvement of the deposition process, which was not yet suitable for mass productions. The »Institute for Microsensors, -actuators and -systems« (IMSAS) uses micro-structured layers of nickel, nickel alloys, gold, silver and copper to produce micro-switches, -valves, -pumps, -coils and -gyroscopes. The metal layers put the highest demands on homogeneity, as well as electrical and magnetic characteristics.

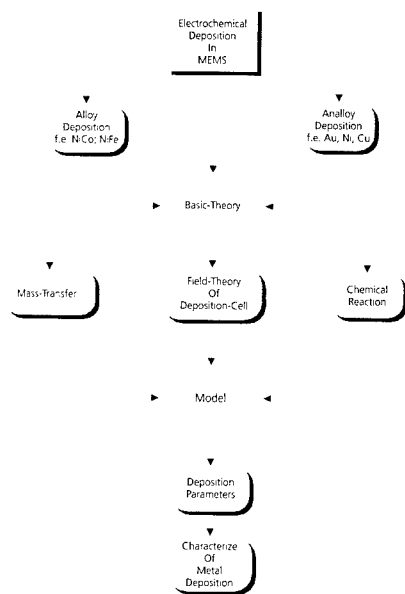


Figure 1:  
Metal deposition in MEMS

In order to reach the desired layer quality, the deposition process has been modelled. The model comprises distinct sub-processes (fig. 1). For the application of deposition processes in microsystem technology several requirements, due to the miniaturisation, have been considered.

Starting point was an electrolyte, which was developed in co-operation with Blasberg-Enthone-OMI. Cyclic voltammetry and electrochemical impedance spectroscopy were utilized to characterize the electrolyte. It has been shown that the mass-transfer to the cathode fundamentally depends on the current density. These effect could be accurately controlled by using pulsed currents.

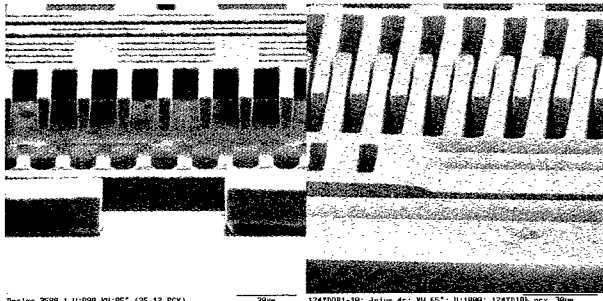
The result of the controlled deposition process is shown in fig. 2 and 3. The minimization of inner stress within movable nickel structures serves as an example to demonstrate the quality of the deposition process.

Fig. 3 shows finger-like movable electrodes of a gyroscope, manufactured with the optimized process. Fig. 2 shows the same device, manufactured with a »standard« deposition process. Due to the process induced inner stress, the movable electrodes are bend upwards.

## Fabrication Facts

plating materials	gold, nickel, copper, silver and nickel alloys
microform material	positive photoresist 3D-UV-microforming up to 100 $\mu\text{m}$
rate of deposition	10 $\mu\text{m}/\text{h}$ dependent of plating materials
resolution	dependent of microform up to 2 $\mu\text{m}$
max. process temperature	50 $^{\circ}\text{C}$ dependent of plating materials
metal parameters	on demand

This technology process has the potential to manufacture with a broad area of economically application and IC-compatible sensors and actuators. The development and standardisation of the separation process takes place at IMSAS in co-operation with the industry to ensure applicability.



Design 3588\_1 U:088 KM:85\* (35\_12.PCX)

Figure 2:  
Ni-Layer within inner stress

324T0001-18: drive 4c: 8M 65\*: U:1088: 124T010b.pcx 30µm

Figure 3:  
Ni-Layer without stress

## Corrosion protection with conducting polymers: mechanism, stability and durability of the protective component<sup>1</sup>

Lux, F.A.

*This work was done at the Intelligent Polymer Research Institute, Wollongong University, Australia*

Intrinsically conducting polymers (ICPs) have been discussed in recent years as corrosion protection materials. State-of-the-art of corrosion protection tells that such materials might be either chemically or electrochemically active as a protecting material. Explanation for chemical activity is pretty obvious, because of the structural resemblance of conducting polymers, like polyaniline, polypyrrole and polythiophene derivatives, with state-of-the-art (low molecular weight) corrosion inhibitor molecules. On the other hand, electrochemical corrosion protection of conducting polymers is discussed in context with so-called active/passive metals and limitation of such activity is to the (doped) electrically conductive versions of conducting polymers. Basically, electromechanical corrosion protection means passivation of the corroding metal surfaces, i.e. anodic stimulation of metal dissolution until the critical corrosion rates of the metals are exceeded, accompanied by the formation of a very thin (10 nm for steels) protective metal oxide layer. Accordingly, the conducting polymers would produce only increased metal dissolution, if the composition of the conducting polymer does not allow for a mechanism to form the protective oxide layer at some critical dissolution (corrosion) rate. Thus, it is clear today, too, that the conducting polymers can not impose electrochemical corrosion protection on its own, but rather only in combination with organic/inorganic auxiliary substances. Accepting this, one would be able to create promising corrosion protection materials, which are environmentally very promising what concerns substitution of lead and chromium(VI) pigments. However, other basic physico-chemical properties of most of the state-of-the-art conducting polymers, in particular the polyaniline and polypyrrole families, oppose the latter conclusion. So conducting polymers normally does need acidic conditions to keep its electrically conductive state alive. The need to keep conductive polymers at low pH-value has serious impact on potential corrosion protection processes. First, corrosion protection with such materials takes place in the heavily acidic regime, where normal oxygen corrosion is superimposed by hydrogen corrosion. Second, oxygen and other molecules get extraordinary high oxidation power under acidic conditions. Third, drying up an acidic solution of pH = 1 (i.e. the wet corroding metal/conducting polymer interface) automatically means increase in concentration of that acidic solution, ending up with concentrated acids and mixed acids, with even higher oxidation strength. In conclusion, oxidation stability of the respective conducting polymers under acidic corrosive conditions becomes a key issue of the whole application. Another important issue concerns the protection mechanism

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involved. The current work focussed on the evaluation of the chemical/electrochemical stability of doped conducting polymers under corrosion protective conditions and identification of the underlying protection mechanism. Resulting is the mechanistical explanation on how oxidatively stable conducting polymers effect electrochemical corrosion protection.



## Sol gel derived $\text{Pb}(\text{Zr}_{0.53}\text{Ti}_{0.47})\text{O}_3$ thin films for MEMS applications

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PZT thin film is the target of the interest in micro electromechanical system, memory etc. The electrical properties of PZT thin films are influenced by the preferred orientation. The control of preferred orientations in sol gel derived PZT thin films and their effect on the electrical properties of PZT capacitors were investigated. The PZT thin films were fabricated using the addition of 15 mol% excess of Pb to the starting solution and spin coating onto Pt/Ti/SiO<sub>2</sub>/Si, substrates. The crystalline phases as well as preferred orientations in the PZT films were investigated using X-ray diffraction analysis (XRD). The microstructure of the PZT films were studied by optical microscopy (OM) and scanning electron microscopy (SEM). The Au/Cr film was deposited by sputtering as the top electrode. The P-E hysteresis loop of these films were measured using a standardized ferroelectric test system which uses the principle of the Sawyer-Tower circuit. The dielectric constants and loss values of these films were measured at 1kHz using an impedance analyzer. The temperature of pyrolysis of the PZT films was found to influence the preferred orientation of film: lower temperatures (250 °C) favored a (111) orientation, whereas higher temperatures (400 °C) favored a (100) orientation. The remnant polarization and the coercive field of (100) orientated films were 14.81  $\mu\text{C}/\text{cm}^2$  and 35.74 kV/cm, while the dielectric constant and loss value measured at 1kHz were approximately 1200 and 0.04, respectively. In contrast, the remnant polarization and the coercive field of (111) orientated films were 22.53  $\mu\text{C}/\text{cm}^2$  and 45.18 kV/cm, while the dielectric constant and loss value measured at 1kHz were approximately 1100 and 0.04, respectively. The PZT films with the (111) preferred orientation show better ferroelectric properties.

**Keywords:** lead zirconate titanate (PZT); sol-gel; preferred orientation; Microstructure; X-ray diffraction, electrical properties

## Temperature dependence of Young's modulus and degradation of CVD diamond

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Temperature dependent measurements of Young's modulus were performed for the first time on black and transparent bulk material of CVD diamond by a dynamic three point bending method in a temperature range from  $-150\text{ }^{\circ}\text{C}$  to  $850\text{ }^{\circ}\text{C}$ . The CVD specimens correspond to a calculated room-temperature Young's modulus of randomly oriented polycrystalline diamond of 1143 GPa. A lower Young's modulus of polycrystalline diamond is caused by crystal imperfections and impurities. At temperature between  $-150\text{ }^{\circ}\text{C}$  and  $600\text{ }^{\circ}\text{C}$  (black type) or  $-150\text{ }^{\circ}\text{C}$  and  $700\text{ }^{\circ}\text{C}$  (transparent type) the Young's modulus is only slightly temperature dependent and decreases monotonically with an average temperature coefficient of  $-1.027 \times 10^{-4}\text{ K}^{-1}$ , which is much higher than theoretically expected. At higher temperatures the bending stiffness and apparent Young's modulus of the diamond beams are drastically reduced to one third of the initial value before fracture occurs due to oxygen etching effects in air. The onset temperature of this degradation phenomenon and the rate of decline are dependent on grain size, texture and the crystal lattice imperfections of the CVD diamond material.

## Yield issues with dicing of MEMS devices

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For more than 20 years, LLNL has been using bulk micromaching, principally in silicon, to fabricate devices that enable numerous measurement and control systems for the programs at Lawrence Livermore National Laboratory. Several devices are typically microfabricated in parallel on each substrate wafer, and separation into isolated components is normally required prior to incorporation into the system. Naturally, we desire the highest possible yield of devices per substrate. The separation process is often performed via precision sawing or »dicing«, and this presentation will discuss the preparations that are necessary as well as the clean-up operations that are required when we using dicing. This presentation will also describe some situations in which we found dicing to be unacceptable.

This work was performed under the auspices of the U.S. Dept of Energy by Lawrence Livermore National Laboratory under contract no. W-7405-ENG-48.

**Keywords:** MEMS, Microtechnology, Instrumentation

## **MEMS in portable applications**

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Over the last ten years, LLNL has been developing microtechnology for instrumentation with particular interest in field applications for the biomedical sciences and environmental monitoring. In general, we operate in the »application-pull« mode, rather than the »technology-push« mode. We have generally found that fabrication techniques in the »mesoscale« range (larger than »microscale«) have been the most enabling for our applications. This presentation will use examples from flow cytometry, polymerase chain reaction, gas chromatography, and electrophoresis.

This work was performed under the auspices of the U.S. Dept of Energy by Lawrence Livermore National Laboratory under contract no. W-7405-ENG-48.

**Keywords:** MEMS, Microtechnology, Instrumentation

## Characterization of transition metal oxides by ultrasonic microscopy

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Transition metal oxides are promising candidates for potential use in sensor applications. Recently they attract again some attention due to their unique electronic transport properties dependent on temperature and the field strength of external magnetic fields. From the scientific point of view the deeper understanding of the complex mechanisms operating in transition metal oxides pose a challenge to solid state physicists. The increased activity in basic research therefore is probably the main reason for renewing one's efforts in crystal growth of these materials and for some progress in the production of larger single crystals, which has been achieved in the meantime. Depending on the technique and preparation conditions chosen, the quality of the obtained crystals, however, often suffers from segregation processes and the spontaneous formation of structural domains during crystal growing. Owing to their widely spread distribution, minority phases of a few percent are hardly detectable if ever by X-ray diffraction. Furthermore, most of the conductive oxides are opaque and thus investigations performed by means of optical methods are limited to surface regions only. In such cases high-resolution acoustic imaging techniques may serve as efficient non-destructive tools for characterizing and evaluating the crystal quality. Here we present examples of the successful use of ultrasonic microscopy concerning this issue. In particular, we report on investigations of single-crystalline  $\text{VO}_2$ , which shows a sharp metal-insulator transition near ambient temperature and of Sr-doped  $\text{LaMnO}_3$ , which is a mixed-valence perovskite with colossal magnetoresistance.

## Stress and fatigue life modeling in thin attach layers

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The use of solid state modules for power conversion and distribution has the potential to significantly improve the efficiency and performance of power electronic products. Before these modules will achieve widespread use, however, packaging must be developed that will ensure reliable operation of the modules under harsh environmental and operational loading conditions. Reliability can be significantly improved by employing the physics of failure methodology during module design. The physics of failure methodology models the reliability of electronic modules based on an understanding of the fundamental chemical, mechanical, electrical and thermomechanical root cause mechanisms by which electronics fail.

One of the major sources of failure in power modules is cyclic thermo-mechanical shear stress in the solder attach layers caused by power switching and ambient temperature cycling. In this paper, physics-of-failure analysis is used to generate a comparative analysis of the stress present in the attach layers of a typical wire bonded module with a single sided integral heat sink structure versus that present in the attach layers of new module having a double sided integral heat sink structure. Physics-of-failure models used in the analysis will include an existing fully elastic multilayer stress model and a unique viscoplastic stress model developed at our research center. The predictions of these two models will be compared to a third prediction obtained using Finite Element Modeling. Damage models relating the relative stress levels to time to failure in the field will also be discussed along with experimental techniques to determine the constitutive and damage accumulation properties of thin film attach materials.

This paper will also present details of an ongoing initiative at our research center to incorporate the physics of failure methodology into a software system for conducting multi-objective design optimizations. Such optimizations are necessary as a design based solely on minimum stress in the attach layers may result in the acceleration of failure by competing mechanisms. Furthermore, the solutions that provide the maximum reliability may not optimize the design with respect to cost, thermal or other considerations. The viability of incorporating different models for attach fatigue into this program will be discussed. The software system consists of a reliability assessment program, a thermal analysis program, a cost analysis subroutine and an optimizing routine used to manage the engineering tradeoffs. Further, all the systems are united by a commercially available interface. This tool will, for the first time, permit computer-aided multiobjective design optimizations of electronic products.

## A resonance method for the determination of Young's modulus and residual stress of thin microstructures

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Accurate knowledge of mechanical properties is an essential issue for the design of micromechanical systems and necessary to monitor technological process uniformity and repeatability. While the mechanical behavior of bulk material is well studied there is a lag for thin films materials. It is known [1] that Young's Modulus and especially the residual stress of thin films are strongly influenced by film thickness, deposition parameters (temperature, pressure etc.) and subsequent high temperature processing steps. Both material properties affect the stiffness and natural frequencies of the device, multilayer structures bend out of shape and buckling effects may occur. Traditional methods for the characterization of bulk materials are hardly applicable on structures with micrometer and submicrometer thickness.

Mechanical properties of thin films are successfully determined by ultrasonic puls-echo methods, nanoindenter techniques, force-deflection or pressure-deflection measurements, resonance methods, electrostatic pull-in measurements, Euler-buckling observations and indicator structures [2-4]. A comprehensive review and discussion of their applicability to the mechanical characterization of thin films are given in [5].

The goal of the presented approach is to extract Young's modulus  $E$  and residual stress  $s_f$  simultaneously on a series of test structures. Therefore an array of cantilevers and on both sides clamped beams with variable length are etched into the layer material. Samples are stimulated electrostatically to small signal oscillations and their resonance frequency is measured by a Laser-Doppler-Interferometer. According to the non-linear beam theory the stiffness and consequently their resonance frequency is a function of the bending energy described by  $E$  and the string energy described by  $s_f$ . The variation of beam length allows to separate both terms by nonlinear function fitting. The advantage of this approach is that measurements can be made in-situ using ordinary vibration monitor equipment.

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## Preparation of micro patterns with profile heights up to 100 $\mu\text{m}$ by sol gel technology

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A new synthesis and processing route for micro patterns with heights up to 100  $\mu\text{m}$  by gel embossing has been developed. For this reason, organically modified nanoparticulate Nanomer<sup>®</sup> sols prepared by catalysis of glycidylxypropyl-, metacryloxypropyl-, methyl-, phenyl- functionalized silane, tetraethoxysilane and titaniumisopropylat in combination with colloidal silica or boehmite sol was used.

Wet gel coatings with thickness up to 150  $\mu\text{m}$  are obtained by dipping or spinning of glass, polymers and Si-wafer substrates. After a predrying step, micro patterns are obtained by embossing using pressures of only 0,3 N /  $\text{mm}^2$ . The embossing properties of the wet gel have been adapted to different pattern heights by variation of the chosen alkoxid precursors, the solid- and solvent content.

Low cost and flexible silicon rubber stamper are used to avoid adhesion between the embossing stamper and the coating. During the embossing process, the structures are fixed depending on sol composition by thermal treatment or UV radiation. After the patterning step, the structures are densified at temperatures from 80 °C up to 500 °C. The linear shrinkage of the micro patterns depends on the used composition and is limited to about 25 % for glasslike structures densified at 500 °C and 1 % for Nanomer<sup>®</sup> structures densified at 80 °C. Other film properties like refractive index, transmission, thermal and mechanical stability are also variable. Nano- and microstructures with sharp edges can be obtained as shown by high resolution secondary electron microscopy. Sharply edged bond and command structure of 100  $\mu\text{m}$  in height, light trapping structures with pyramids of 7  $\mu\text{m}$  in height and 10  $\mu\text{m}$  in width on an area of 20 mm x 20 mm, hologram structures on an area of 100 mm x 100 mm and fresnel lens with a relief height of about 1  $\mu\text{m}$  have been fabricated by this method.

## Non-destructive characterization of ferromagnetic layers and layer systems

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Ferromagnetic layers have gained increasing interest in the sector of microsystem technology. On the one hand magnetic layers are applied for sensors and actuators, on the other hand extremely thin layers with the thickness of only some nm are being developed for applications in magnetoelectronics for M-RAMs and M-Transistors. Therefore, high interest has arisen towards the non-destructive characterization of these layers concerning mechanical and magnetic properties as well as residual stresses. There is a need for further methods, characterizing the dynamic behaviour of the magnetic field, e.g. processes occurring during magnetization reversal and magnetic coupling.

At the Fraunhofer Institute for Nondestructive Testing in Saarbruecken different methods for the characterization of ferromagnetic materials, particularly ferromagnetic layers, with medium and high resolution are being developed and tested. In comparison to the commonly applied methods for the imaging for ferromagnetic structures these techniques are especially sensitive to dynamic remagnetization behaviour and magnetic properties and structures below the covered surface. No special preparation techniques are needed.

The paper presents the Eddy current and Barkhausen microscopy and the photothermally modulated stray field technique in comparison to other microscopic techniques by means of several examples. The characterization of amorphous magnetic materials such as soft magnetic layers, which are applied in data storage systems, will be demonstrated.

## Fracture electronics and thermo-mechanical compatibility (TMC) of microcomponents in high tech systems

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**Thermo-mechanical compatibility (TMC)** of different microcomponents in electronic and mechatronic systems has become more and more important in the recent years. It is not at all a simple procedure to guarantee the traditionally high reliability standard in e.g. automotive electronics and other high tech applications using low cost packaging technologies (e.g. CSP) without taking into account the problems caused by the so-called effect of »thermal misfit« or »thermal mismatch«.

In very simple applications only it is possible to give a suitable solution by means of conventional design rules based on pure elastic or visco-elastic simulation tools.

More advanced systems require to take into account thermal fatigue, creep and effects of local plasticity and their coupling as well. Nonlinear effects (e.g. the creep behaviour in the high temperature automotive electronics applications, in power semiconductor applications etc.) lead to more complicated material behaviour both regarding the constitutive material equations and the coupling with geometric parameters (e.g. layer thickness, dimension of solder regions). The author is going to deal with this kind of problems taking into account both theoretical and experimental aspects.

Examples described in the talk concern automotive sensor systems. New concepts of thermo-mechanical compatibility for microcomponents in high tech systems have to take into consideration improved design solutions for crack avoidance measures as well as coupled testing and simulation tools for advanced reliability estimations. This will be shown in more detail for advanced microsensor packages for automotive applications. Finite element calculation and microdeformation measurements have been coupled to receive improved input data for advanced reliability analysis which has been achieved taking into account additional information obtained by means of acoustomicroscopy (to detect delamination effect of plastic encapsulation and related underfiller defects as well), laser scanning microscopy and microDAC image correlation technique.

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## Fabrication and characterization of plastic (PMMA) for inexpensive MEMS components

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Several applications require increasingly cheaper sensor and MEMS components. In some cases more expensive materials can be substituted by more inexpensive plastic materials. It should be also possible to fabricate bulk micromachined elements and sensors using a fast and cheap fabrication technique. A combination of these structures with semiconducting polymers would allow the fabrication of low cost sensors when compared with silicon sensors.

In a first step we have prepared plastic diaphragms by a hot embossing technique using a stainless steel stamp. In this work we discuss the quality in terms of membrane thickness and surface roughness and reproducibility. Furthermore we will report on Young's modulus and other mechanical parameters of this material.

Especially the elastic and plastic behavior of PMMA under varying temperature conditions will be discussed.

## Microproduction technology – key for micromaterials, innovative products, and new markets

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The frame work programme »research for production of tomorrow« of the German Federal Ministry for Education and Research (BMBF) has been started since October 1999, and is managed by the Project Management Agency for Production and Manufacturing Technology Programmes (PFT) at the Research Centre in Karlsruhe.

There are four key actions in this federal programme:

- markets and strategies in product design,
- technologies and production equipment,
- new ways of collaboration for manufactures,
- workers and management of change in enterprises.

Within the second key action of production equipment there is a main stream of industrial microproduction technology. Miniaturisation of products or components needs more reliability in manufacturing processes with micro materials for new products. Examples are the assembly of electronic devices for telecommunication, the economic treatment of smart materials like shape memory alloys for stents in health care, or harnessing light to produce mico tools for stamping and embossing of electrical micro connectors.

The goal within this part of the work programme »research for production of tomorrow« is to improve production and test equipment, to strengthen the skills of workers for new production processes, and to have an impact for manufactures of electronics and micro products towards more reliable and efficient ways of manufacturing innovative products for markets of tomorrow. Means of this information and funding campaign are collaborative research and development projects, technology transfer activities and experience exchange in industrial working groups.

Coordination with other national programmes like for laser and micosystem technologies, new materials and information technology is important to use synergy in prototyping new materials or technical systems on one hand, and on the other hand, getting these materials and systems implemented into flexible and economic manufacturing of new products.

On international level the German initiative is coordinated with the fifth frame work programme of the European Community in the field of »Innovative products, processes and organisation (IPPO)« and EUREKA- FACTORY, the initiative for technological development in »Factory for the future« for whole Europe, including small and medium sized enterprises in Eastern Europe.

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## Study of thermal deformation in underfilled flip-chip packages using high resolution Moiré interferometry

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Thermally induced stresses play an important role in controlling the structural reliability of flip-chip packages. As the demand for increased performance continues to shrink solder bump pitch and size, it becomes increasingly difficult to measure thermally induced displacements and strains within these packages. To address this issue, we have developed a high-resolution moiré interferometry system based on the phase shift technique to map thermally induced displacements and strains in underfilled flip-chip packages. This system was demonstrated using a thermally loaded underfilled flip-chip package with 250mm solder bump pitch. The results revealed a resolution of 26nm in mapping the displacement contours and a sensitivity to measure thermal displacements within features as small as 10 microns. The resolution corresponds to 16 times greater than conventional moiré interferometry, making it possible to accurately determine displacements and strains within features normally contained within one moiré interference fringe. Image analysis based on a variational principle has been developed to extract deformation and strain distributions. This was used to extract stress-intensity factors at the die corner using experimental data and compared with results from finite element analysis.

## Submicrometer notch machining by FIB and fracture and fatigue behavior in silicon microelements

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In order to develop a reliable micromachine in a service operation, much care must be taken not only to processing methods but also to micromechanical evaluation, i.e., mechanical properties of mm sized microelements including fatigue and wear. One of the most important issues to be considered is the influence of notch and fatigue loading on fracture strength in microelements in different environments. In this investigation, the influence of notch and water environments on the quasi-static and fatigue fracture behavior was investigated in single crystal silicon microelements. The tests were conducted in smooth microcantilever beam samples and notched ones. Smooth single-crystal Si microcantilever beam specimens were prepared by micromachining (photo-etching) of (110) silicon wafers. For some specimens, small sized notch was machined 100  $\mu\text{m}$  away from the sample root by using a focused ion beam system. An adequate machining condition was investigated: the shape of machined notch was evaluated by an atomic force microscope (AFM). The radius of the curvature of the notch measured by the AFM decreased with an increase in notch depth, and it ranged from about 20 nm to 100 nm. In order to perform quasi-static and fatigue tests, a specially designed testing machine developed by the authors was employed: small loads are applied by means of an electromagnetic actuator. Single-crystal Si microelements deformed elastically until final failure, giving a brittle nature. The maximum fracture strength of a microcantilever specimen reached about 7.7 GPa, which was still higher than that obtained in mm sized samples. In addition, the fracture strength decreased with an increase in notch depth, although the notch depth was in the order of sub-mm. This means that sub-mm order notch, which is often regarded as such roughness for ordinary sized mechanical parts, caused a decrease in the fracture strength of Si microelements. The fracture initiated at the notch, and then propagated in the direction normal to the sample surface in {111} plane.

Fatigue tests were also conducted in laboratory air and in pure water at a stress cycle frequency of 0.1 Hz and a stress ratio of 0.1. In laboratory air, no fatigue damage was observed even though the surface was nanoscopically examined by the AFM. Dramatic effects were observed when the fatigue tests were conducted in pure water. The fatigue lives in water were decreased: crack formation in {111} plane was promoted by a synergistic effect of dynamic loading and water environment. Atomic force microscopy was capable of imaging nanoscopic cracks in {111} plane, which caused the failure in water. This means that environmental effects, in particular water environments, are extremely important in strength in micromaterials.

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## BaTiO<sub>3</sub> - Ceramics cluster intergranular impedance model

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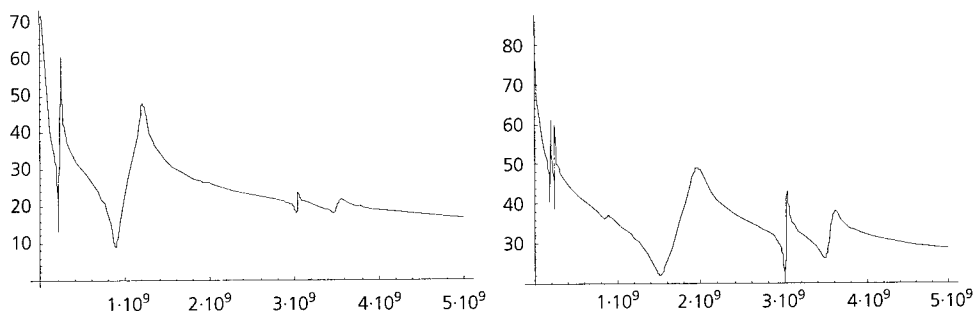
The significance of ceramics materials increases in general electronics. There is growing interest in high-performance electronic ceramics components for modern communications, signal processing, computers, power transmission and control system use. Therefore it is important to increase scale of integration of ceramics components within a ceramics integrated circuits.

The aim of this paper is to improve tools and methods for better prediction of ceramics IC properties. With this goal we research the intergranular impedance. The basic idea came after the results that consider intergranular capacitance. Moreover, in this paper we spread our objective to intergranular impedance. Actually we suppose that between each two contacted grains there is an elementary intergranular impedance,  $Z_e$ , that consists of a serial RLC in parallel with a capacitance  $C_1$ . In s-domain  $Z_e$  can be expressed in symbolic form as

$$Z_e(s) = \frac{1 + CR \cdot s + CL \cdot s^2}{(C_1 + C) \cdot s + C_1 CR \cdot s^2 + C_1 CL \cdot s^3}.$$

The contact area between two grains can be viewed as a planar condenser with capacitance  $C_1$ . Therefore the dominant effect in  $Z_e$  at low frequencies comes from capacitance,  $C_1$ . However at higher frequencies the effect of serial oscillator circuit can not be neglected.

Thinking about a ceramics sample as a complex structure made of many contacted grains, we consider it as a complicated interconnection of elementary impedances. Finding the equivalent impedance of the sample based on  $Z_e$  seems to be too much complicated. However it is known that grains tend to form clusters. Hence, we suggest a hierarchical approach. Firstly we intend to determine equivalent impedance of a cluster,  $Z_c$ , with the aim to approach to higher levels of abstraction in our subsequent research.





The support for this idea we found in the fact that results obtained for  $Z_c$  (for more than two grains) by symbolic simulation are similar to results measured on ceramics samples.

Actually, in this paper we consider a cluster formed by five grains. This gives a hexahedron formed by two pyramids with a common triangular basis where the grains are placed as nodes. They make nine intergranular contacts. For randomly chosen parameters for nine different  $Z_e$  we obtained results presented in the Figures below. The left figure presents normalized magnitude of  $Z_c$  when opposite peaks of pyramids were chosen for ports, while the another shows  $|Z_c|$  for the same cluster when the equivalent impedance is looked between two nodes from the basis.

## The application of cluster intergranular impedance model for different BaTiO<sub>3</sub> - ceramics cluster structures

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The problem of new ceramics material prognosis is very important from the point of ceramics electronic properties design. Therefore we intend to contribute to the development of methods for recognition electronic parameters in grain boundaries contacts.

We assume that every two grains in contact form an elementary intergranular impedance that can be modelled with circuitry depicted in Figure 1. The magnitude of the elementary impedance,  $|Z_e|$ , as a function of frequency is given in Figure 2 for one set of parameter values, while Figure 3 shows this magnitude for different parameter values extracted from measured BaTiO<sub>3</sub>-ceramics sample.

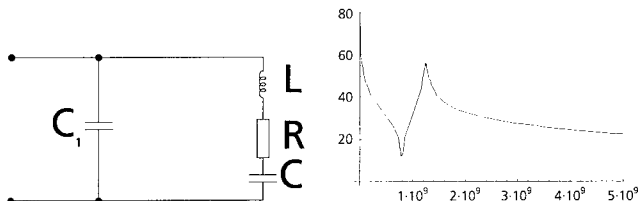


Figure 1

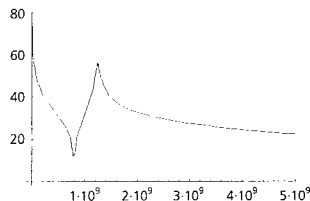


Figure 2

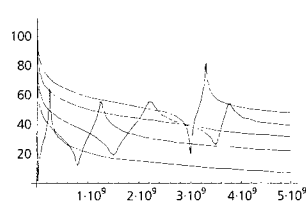
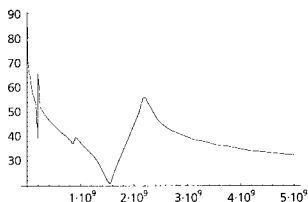


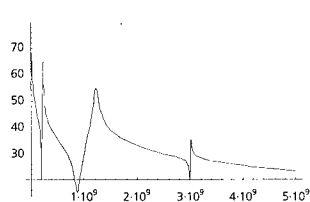
Figure 3

One ceramics sample consists of many grains mutually contacted. As SEM micrographs verify, the grains usually form clusters. The minimum cluster contains two grains and one contact. However, three and more grains can form different combinations of contacts. Three grains make a cluster with three contacts, i.e. with three interconnected elementary impedances. If equivalent impedance of the cluster,  $Z_c$ , is assumed to be a two-port, it can be viewed as a parallel connection of one  $Z_e$  with serial connection of two other. There are three combinations for  $Z_c$  depending on chosen ports.

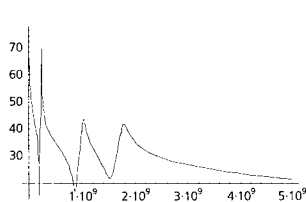
This paper is aimed to verify the idea of intergranular impedance model as depicted in figure 1 comparing results obtained by symbolic circuit analysis of equivalent cluster



Three clusters:  $Z_c = Z_{e1} + Z_{e2} \parallel Z_{e3}$



Four clusters:  $Z_c = Z_{e1} \parallel Z_{e3} + Z_{e2} \parallel Z_{e4}$



Four clusters:  $Z_c = Z_{e1} \parallel Z_{e4} + Z_{e2} \parallel Z_{e3}$

impedances with results obtained by measurements on real  $\text{BaTiO}_3$  ceramics samples. Different interconnections of elementary impedances, that correspond to clusters formed by three and four grains are considered. The obtained results undoubtedly confirm proposed cluster impedance model. Some of the results are presented in the figures below.

## Stability of flat boundary under surface diffusion

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The problem of a flat surface stability under surface diffusion was studied in 1972 by Asaro D.G. and Tiller W.A. first. It was considered the elastic stress body occupied a half-plane with a free bound subjected to an action of surface diffusion. In 1998 A. Danescu and F. Sidoroff studied this problem in the case of the rigidly fixed strip. In the present work the case of different elastic constants of the strip and the substratum is considered. The elastic infinitely long material consists of the substratum and the surface layer. Its upper surface is free, but one is subjected to an action of surface diffusion. In addition, the surface layer is subjected to an action of the permanent stress in direction parallel to the free surface. It is assumed that a some perturbation is given on the upper surface. The aim of this study is to establish a dependence of the free boundary stability on the layer thickness, the wavelength of the curved surface and elastic constants.

## Future developments in electrically conductive adhesive technology

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The paper will review past accomplishments in the development of robust electrically conductive adhesive (ECA) materials, processing technologies, and applications. With the current status as a springboard, it will proceed to examine current problems, including gaps in our understanding of technology fundamentals. This analysis will lead in turn to near-term predictions of research activities to be undertaken to address the problems identified. The presentation will concentrate on isotropic ECAs, and will include some long-term speculations on innovative variations of current technology.

## Materials properties with respect to microsystem technologies

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The technology of microsystems allows the integration of electronic, acoustic, mechanical, thermal, optical and magnetic functions within one product. The tendency to a continuous miniaturization and the corresponding increase in the density of integration is an increasing challenge to the materials in use. Due to the multifunctional demands a wide variety of materials are being applied including semiconductors, insulators, metals and alloys, polymers as well as special functional materials. Since these material combinations are far removed from thermodynamic equilibrium there is a strong tendency for phase transformations and intermixing at elevated temperatures. Often, extreme service conditions prevail such as high temperatures, aggressive surrounding media, electromigration at high current densities which require excellent contact resistance on long time scales, good adhesion between the metallic circuits and the substrate, negligible fatigue, good creep resistance of conducting interconnects and bonding materials, good corrosion / erosion resistance in an aggressive environment etc. As such, the materials specific properties of the different materials in use are important parameters in optimizing microsystems and improving their lifetime. These include mainly electronic (electrical conductivity), thermal (specific heat, thermal conductivity, thermal expansion) and mechanical (elastic, plastic, viscous flow, creep) properties.

As the outcome of some of the recent research activities in Germany, a consortium of different small and large size companies and different research institutions has been formed. One goal of this project name SiMiKo (**S**imulation of **M**icrosystem **C**omponents) is the establishment of a data bank for temperature dependent materials properties for microsystems. These reliable data will then be fed into commercial finite element simulation programs. In addition, certain particular unknown materials properties will be experimentally determined and added to the data bank under consideration of the sample preparation which can lead to either amorphous, nanocrystalline, microcrystalline or single crystalline (epitaxial) structures. Furthermore, new models for the thermomechanical materials properties are being developed with the consideration of the micro / nanostructure, reduced sample size, defect density, internal and thermally induced stress and the role of internal interfaces. The integration of new specially tailored materials with particular functional properties will lead to a considerable improvement of the lifetime and reliability of microsystems and the development of devices resistant to high temperatures, high pressures and chemically aggressive environments.

An overview will be given on the relevant thermomechanical properties of the different

materials being used and the time / temperature dependent measurement and modeling of these properties for materials with reduced dimensions and confined microstructures. Several examples and their critical issues will be discussed with respect to high power electronic devices, high temperature / high pressure sensors and surface active wave devices.

## Synthesis and optical properties of diamond crystals

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It is well known that physical properties and crystalline quality of synthetic diamond crystals are affected by different impurities (nitrogen, nickel, silicon etc.), defects and internal strains. Therefore the study of the nature of crystal defects would be required for further improvement of the growth process. In this work we give the results of the complex study of the optical properties of the diamond single crystals obtained in various growth conditions. Diamond crystals up to 6 mm in size (1 ct. weight) were grown by the temperature gradient method with the "split sphere" high pressure apparatus. Synthesis was carried out in the pressure and temperature interval 5.3 – 5.6 GPa and 1450 – 1550 °C respectively. The high-purity graphite as the carbon source and different alloys Fe-C, Fe-Ni-C, Fe-Co-Ni-C as the solvent-catalysts have been used in our experiments. For eliminating the nitrogen impurity different elements (Ti, Al, Si) were used as the nitrogen getters. The infrared and ultraviolet absorption and cathodoluminescence measurements have been used for the characterization and identification of synthetic diamonds. The experiments show that the synthetic diamond singly crystal can be classified as Ib or lab types. The crystals with the low nitrogen concentration may be attributed to the pure Ib type. It was found that the concentration of the nitrogen substitutional atoms is different for various growth sectors. The total nitrogen concentration in the investigated samples is in the range 0.2 – 200 ppm and depends on the chemistry of the solvent-catalyst. A number of vibronic bands with zero-phonon lines (1.401 eV, 1.682 eV, 2.156 eV, 2.559 eV, 3.19 eV) have been observed in the cathodoluminescence spectra of the synthetic diamonds grown using Ni alloy solvent-catalysts. The presence, of some of these luminescence centers depends on the nitrogen content in the synthetic diamonds. So, optical spectroscopy studies show that they are very fruitful means for the determination of the nature of different defects and impurity-related complexes in the synthetic diamond single crystals.



## New MoCu and WCu MIM-materials designed for heat dissipation purposes in RF packages

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RF modules in modern radar and communication devices used for industrial or automotive sensors and mobile communication networks are designed to work at frequencies in the mm-wave range between about 10 and 100 GHz with increasing tendency. Partly, high amounts of heat loss energies produced in extremely small areas of the GaAs chips, which are required for rf frequency circuits, have to be dissipated and drained. Materials for packaging such a kind of modules must offer the following features: appropriate strength, high thermal conductivity, low thermal expansion, and a high-precision workability at low-cost level.

Early investigations performed in a project called »New Metallic Material Concepts for mm-Wave Functional Packaging«, sponsored by the German Ministry of Research, Education and Science, resulted in feedstocks made from molybdenum and copper powders, completed by waxes and polyolefines, which after injection moulding, debinding, and sintering yielded MIM parts having some poor properties as inhomogeneity, porosity, or faults in geometry. Step by step they could be improved, so that recent developments resulted in parts showing very fine, homogeneous microstructures with high density. Moreover, the geometry is expected to become almost perfect.

In addition to the practical work there also had to be performed theoretical considerations about the thermal properties and residual stresses of the packages. Several materials for the device casing (CuMo, WCu, Kovar) have been compared. This was achieved by thermo-elastic finite element modelling. The packages include a LTCC substrate - soldered onto the MIM part - on which the heat sources are placed. Two situations were analysed: the mating state where the assembly is put together at 230° C and cooled down to room temperature and the operation mode where the solder layer has relaxed to equilibrium at room temperature and the heat sources are switched on.

We find for the mating state that Kovar and W<sub>90</sub>Cu cause the smallest stresses due to the similarity of their CTEs to that of the substrate. Care has to be taken of the large tensile stresses in the solder layer. In the operation mode inhomogeneous heating produces different effects. Here, materials having a higher thermal conductivity are favourable (e.g. W70Cu or MoCu).

**Key figures:** heat sink, material development, metal injection moulding, thermo-mechanical analysis, finite element method

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## High aspect ratio structuring by ICP etching and metals and ceramics micro forming with powder injection molding

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Micro system technology has been widely spread to various fields such as the fluidic, medical, optical, data storage applications. For the real commercialization of the micro devices, technology development for low cost mass production is strongly encouraged, especially for the bio-medical application of micro system technology, where the devices are expected to be disposable or recyclable.

The above mentioned is the reason why we developed the technology for mass production of metallic and ceramic micro components with micro powder injection molding (mPIM). We can expect the low cost fabrication of three-dimensional micro parts through this technique. In the present work, mold insert was prepared by ICP Silicon deep etching technology, then mPIM was processed using silicon mold insert. Various shapes of mold insert are fabricated and tested. The powder used here includes stainless steel, Titanium and PZT considering applications for medical use ultrasonic transducers, and actuators. Above all, Titanium micro forming is strongly emphasized for the biocompatibility. A multi-component binder system comprising of PAN250 (a patented binder) + EVA (Ethylene Vinyl Acetate) + HDPE (High-Density Polyethylene) was used. Super critical debinding method is applied prior to final sintering process.

Various kinds of micro parts could be structured and formed with high aspect ratio of more than 3. Blind hole and through-hole mold inserts are processed. It was proved that the cavities of blind holes may cause problems during molding because of trapped air in the cavities. We had tried molding after evacuation of the cavities and molding with through hole structure. We have also demonstrated the fabrication of metal-ceramic composite structure by Deep RIE and mPIM.

**Keywords:** Micro injection molding, lead zirconate titanate (PZT), Titanium, stainless steel, Micro system

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## **Ion implantation for micromaterial fabrication - fabrication and material properties**

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We applied the ion implantation method to micro-material fabrication. The ion-implantation which was known as a semiconductor doping or a surface modification technique, is able to modify several micron meters depth of substrate when the energy of ion is MeV. Though, ion-implantation is able to make micro materials for micro-devices. Further, we can make many kind of materials from freedom selection of ion species, ion dose, ion energy, and substrate temperature, etc.

We developed a separation process of the ion-implanted region from substrate using different etch rate. A micro cantilever beam was fabricated by obtained method. A thickness of the micro cantilever beam was 0.7 micro-meter which is enough to micro device. The Young's modulus and surface resistance were measured as its basic properties. For example, the cantilever which was implanted 3.1 MeV gold ion and  $1 \times 10^{17}$  ions/cm<sup>2</sup> dose, shows about 60 GPa and 36 kW (Ohm). The fabricated materials were also observed by cross-section TEM.

We will present and discuss the possibility of the micro materials fabricated by this method.

## Cohesive surface modeling of fracture

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In the traditional approach to fracture mechanics, the stress analysis is separated from a description of the actual process of material failure. A parameter characterizing the crack tip field, e.g. the energy release rate, is assumed to be a material property and known from experiment. Crack initiation is identified with this parameter reaching a critical value and the description of continued crack growth depends on knowing this parameter as a function of the amount of crack growth and the crack speed. Here, an alternative approach will be described where the failure characteristics are embodied in a phenomenological constitutive relation that describes separation along one or more cohesive surfaces. Constitutive relations are specified independently for the material and for the cohesive surfaces. Fracture emerges as a natural outcome of the deformation process, without introducing an additional failure criterion. The characterization of the mechanical response of a cohesive surface involves both an interfacial strength and the work of separation per unit area, which introduces a characteristic length into the formulation. This framework has been used to address issues including void nucleation, quasi-static crack growth, dynamic crack growth, thermal crack growth and reinforcement cracking in metal-matrix composites. The results obtained have reproduced, at least qualitatively, a wide variety of observed fracture behaviors. There is no unified description of these phenomena within a traditional fracture mechanics framework.

Here, some results obtained using this cohesive surface framework are discussed. Examples will be taken from analyses aimed at ascertaining limiting crack speeds and how cleavage can occur in the presence of plasticity. It is worth noting that, according to classical continuum plasticity, the maximum stress attained at a blunted mode I crack tip is of the order of 3 to 5 times the material's flow strength, which is typically too low to cause cleavage. A cohesive surface analysis of crack growth where inelastic deformation arises from the collective motion of discrete dislocations gives rise to a high enough stress level near the crack tip to permit cleavage.

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## Characterization and aging of optical fiber Bragg gratings

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Progress made in the last decade in opto-electronics and glass fiber technology is revolutionizing telecommunications. Wavelength multiplexing was introduced to satisfy demand for ever higher bandwidth. Fiber optical Bragg gratings as passive elements play an important role, e.g., for wavelength stabilization and tuning of lasers, as filter elements in add-drop multiplexers, or for dispersion compensation. On the other hand optical fiber Bragg gratings are very sensitive in wavelength response to stress and temperature making them well suited for sensing applications. They are easy to integrate in structures, electromagnetically immune, allow wavelength domain or time domain multiplexing, and can be used at high temperature and harsh environmental conditions, where electrical sensing schemes have limited performance or even fail.

This paper presents investigations on the optical, thermal, and mechanical properties of fiber Bragg gratings, which are of fundamental interest for assessing and optimizing Bragg grating functionality. Investigation of reliability, durability and long term stability are indispensable for new fiber Bragg grating based devices used in telecommunications, long term monitoring and process control.

For both, sensor and telecom applications optical Bragg grating properties like Bragg wavelength, spectral shape and width, reflectivity, transmission spectrum of reflected core and cladding mode, and polarization dependence have to be known. For sensor applications Bragg gratings have to be tested for their reproducible response to stress and temperature and their immunity to environmental influence.

Optical glass fibers and Bragg gratings can be treated like brittle materials. They are susceptible to stress corrosion leading to strength degradation and mechanical failure at any applied stress. Humidity and temperature accelerate this effect. We used a power-law based mechanical lifetime model to describe stress corrosion of UV-induced Bragg gratings and an Arrhenius-type model to address zero-stress aging.

Refractive index modulation and mean index change of Bragg gratings produced by modulated UV-irradiation of fiber core are described by carriers trapped in various energy states. They decay when exposed to elevated temperature. The resulting wavelength drift with time limits accuracy for a specific sensor filter application. Thermal decay can be modeled with an Arrhenius-type temperature dependence but assuming a broad distribution of activation energies. Decay data was analyzed with a mastercurve approach. The model predicts that Bragg gratings which are pre-annealed at a higher than operating temperature for some time will be much more stable at lower operating temperature, because low energy states are depleted.

In conclusion we characterized different fiber Bragg gratings with respect to their optical, mechanical, and thermal properties and will demonstrate their reliability and durability under various service conditions as experienced in telecom devices and sensing

applications. Changing any fabrication parameter in Bragg grating production, e.g., fiber, writing conditions or procedures, may considerably change model parameters and lifetime predictions.

## Humidity ceramic sensor materials on the basic of $\text{TiO}_2$

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One of the trends in the development of humidity sensors is the use of sensors elements based on ceramic materials. The materials used most often as basic components in the development of ceramic humidity sensors are  $\text{TiO}_2$ ,  $\text{SnO}_2$ ,  $\text{ZnO}$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ , etc. The composition and structure of ceramics has a considerable influence on the sensor parameters and characteristics. They can be controlled by the thermal conditions of synthesizing and using various alloying additions. Considerable interest in the development of ceramic humidity sensors is displayed in  $\text{TiO}_2$ . The influence of different alloying additions, such as  $\text{V}_2\text{O}_5$ ,  $\text{Nb}_2\text{O}_5$ ,  $\text{SnO}_2$ ,  $\text{La}_2\text{O}_3$ , etc., on the parameters and characteristics of  $\text{TiO}_2$ -based ceramic humidity sensors has been studied.

The present paper studies the influence of  $\text{Bi}_2\text{O}_3$ ,  $\text{PbO}$ ,  $\text{CdO}$  and  $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$  as an alloying addition and of the thermal conditions of synthesizing on the characteristics and parameters of  $\text{TiO}_2$  based ceramic humidity sensors. The sensors have been developed using a standard ceramic technology. This have been synthesized in the temperature range  $850^\circ\text{C}$ - $1050^\circ\text{C}$ . The alloying additions in the tested specimens range from 1 to 10 mol%. The influence of the alloying additions and the synthesizing mode of the sensor characteristics, sensitivity, hysteresis and response time of sensors has been studied. An X-ray diffractometry (XRD) has been performed in order to study the structural composition of ceramic humidity sensors. The influence of the ambient temperature on the sensor parameters and characteristics has been investigated as well.



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## Thermographic testing methods with high temporal and spatial resolution

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Applications of high speed thermography with frame rates up to 1 kHz will be demonstrated on examples. By applying special optics a high spatial resolution up to 50  $\mu\text{m}$  is achieved. Passive thermography, this means thermography without additional heating procedures, enables one to study thermal processes in electronic elements, which take place during switch on and switch off processes.

Silicon is transparent for electromagnetic waves in the near infrared range. Therefore the thermographic technique enables a visualisation of structures within the silicon chip.

If the thermographic camera is combined with an additional heating system, for example flash lamps, then one is talking about active thermography or pulsed thermography. This technique is applied to detect local defects, in particular delaminations in layered systems. Due to the high time resolution very thin layers with good thermal and electric conductivity can be tested. Moreover, the high local resolution allows recognition of small defects.

If a spatial resolution of some  $\mu\text{m}$  is required then thermal microscopy can be applied. The thermal response due to the periodic stimulation of the test object by a laser is measured. If the laser spot is scanned across surface of the specimen amplitude and phase images are obtained by applying the lock-in-technique. Thermal wave microscopy is also used to detect small vertical cracks and to estimate thickness, density and thermal diffusivity of surface layers.

## Determination of mechanical parameters of solder materials using the miniature measuring method »small punch test«

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In order to model the aging of SMT (Surface Mount Technology) solder materials, which is a prerequisite for lifetime predictions of microelectronic joints as well as of the microelectronic package as a whole, it is necessary to know the current mechanical parameters of the solder as accurately as possible. The mechanical parameters are understood as basic parameters, such as Young's modulus, yield stress, hardening coefficient or creep parameters as well as damage characteristics (e.g., fracture toughness, energy density to fracture or J-integral).

In fact, the material behavior of solders is strongly affected by a repeated change of the temperature and the associated thermal loads. Consequently, the current, i.e., »remaining« properties of in-service solders are of special interest. SMT applications use only very small amounts of solder. Therefore, the Small Punch Test (SPT) can preferably be used to secure such materials data. The SPT is essentially a miniature version of a deep-drawing test during which a load-deflection curve is obtained. In combination with a FE-simulation the uniaxial stress-strain behavior and the fracture toughness of the solder material can be determined. It seems worth pointing out that by virtue of this methodology use of standard size specimens, which are not always available, is avoided *a priori*.

In particular we will report on the outcome of various small punch experiments in combination with FE studies, which have been carried out for various lead and lead-free solder materials. These were metallurgically examined before and after they had been subjected to well-defined temperature treatments, i.e., aging characteristics.

## R-curve behavior of PZT-ceramics under the influence of an electric field

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The fracture resistance curve (R-curve) of different commercial PZT was measured with compact tension specimens under the influence of an electric field applied parallel to the crack front. A strong influence of the electric field on the starting and plateau value as well as the length of the R-curve was found.

Generally a toughness increase was detected with increasing electric field. The main toughening mechanism is thought to be ferroelastic domain switching to the development of a process zone around the crack.

The toughening effect is estimated from the crack-tip stress-intensity change induced by ferroelastic domain switching near the crack tip using the weight function formalism developed for stress induced transformation toughening of zirconia ceramics. In order to develop a semi-quantitative picture of the toughening effect of the investigated ceramics the polarization and strain hysteresis was measured. The ferroelastic behavior was determined in four-point bending.

## Catalytic application of sol-gel deposited YSZ/Pd,Pt,RuO<sub>x</sub> and YSZ/Pd,Pt,Rh layers on Corning™ catalytic substrates

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The rate of emission of coke particles (PM) by diesel engine can be 7 times greater when engine is cold than in the case of its work at temperatures 500-600°C, after the first ca 120 second of work. Decreasing of PM contents at pointed temperature results from their oxidation, which is relatively quick under that conditions. It is possible to reduce PM contents in exhaust gases at lower temperatures (280-400 °C) by applying oxidizing catalysts.

In the present work the YSZ (yttria stabilized zirconia) layers (mismatch) containing as a active catalytic centers Pd+Pt+RuO<sub>x</sub> or Pd+Pt+Rh nanoparticles, deposited by sol-gel method on Corning™ substrates were studied as potentially useful catalytic materials for reduction of PM emission.

Yttria stabilized zirconia was prepared by hydrothermal treatment of co-precipitated yttrium and zirconium hydroxides. Hydroxides in a gel form were obtained by adding concentrate ammonia water solution to zirconium oxy-chloride water solution containing appropriate amount of yttrium chloride (8% mol as Y<sub>2</sub>O<sub>3</sub>). Hydroxides gel was washed, filtered and than treated hydrothermally at 240°C and 3.3 MPa during 2 hours. Transmission electron micrograph showed that prepared YSZ crystallites have average diameter about 7 nm.

The layer of YSZ on Corning™ substrates was obtained by drying of YSZ nanopowder water suspension films applied on substrates. As prepared samples were impregnated using water solutions containing appropriate quantities of chlorides of palladium, platinum and ruthenium or rhodium. Next impregnated samples were treated either by HCHO or by 10% hydrogen in argon. After reduction the highly active catalytic particles of Pd-Pt-RuO<sub>x</sub> and Pd-Pt-Rh were formed.

The obtained catalysts were tested in exhausting system of diesel engine. It was found that catalytic properties of studied catalytic system are better than that without YSZ layer. YSZ-Pd-Pt-RuO<sub>x</sub>/Rh bed lowers temperature of PM combustion to 350 °C and partially decomposes hydrocarbons. It is supposed that hydrogen *in statu nascendi* from hydrocarbon decomposition aids reduction of NO<sub>x</sub>.

## Degradation of Pt electrodes and micro-heaters of NO<sub>x</sub> sensors

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Conventional NO<sub>x</sub> sensors used for determination of nitrogen oxides contents in spark ignition engines cannot be applied as sensors in diesel engines, because of its accelerated wear during long term exploitation. The degradation of sensors is observed in particular in a central part of catalytic bed in three-way catalyst (TWC). The goal of present work was to explain the cause of accelerated degradation of conventional sensors in diesel engines.

The thick-layer sensor was prepared with active layer TiO<sub>2</sub>-10%SnO<sub>2</sub>-1.5%RuO<sub>x</sub> deposited on a microporous sinters: ZrO<sub>2</sub>-15%SiC and Al<sub>2</sub>O<sub>3</sub>. On the substrate comb-shaped Pt electrodes and inner afterheater made from Pt-Ti powder dispersed in 3Bi<sub>2</sub>O<sub>3</sub>·WO<sub>3</sub> glaze were deposited. In ammonia atmosphere at 800 °C the composite path Pt-TiN-3Bi<sub>2</sub>O<sub>3</sub>·WO<sub>3</sub> of meander shape is formed, which has low temperature resistance coefficient. Such prepared template with electrodes and heater was covered by active layer TiO<sub>2</sub>-SnO<sub>2</sub>-RuO<sub>x</sub>. This active layer was deposited as paste made of powder of TiO<sub>2</sub>-SnO<sub>2</sub> obtained from solution prepared from TiCl<sub>4</sub>·5H<sub>2</sub>O and SnCl<sub>2</sub>·5H<sub>2</sub>O compounds reduced by Ar-10 vol.%H<sub>2</sub>. TiO<sub>2</sub>-SnO<sub>2</sub> layer was next activated by RuO<sub>x</sub> deposited from gaseous phase.

Sensors were installed before catalytic bed inside central part of TWC and after catalytic bed. Tests of sensors life were performed on Zellner engine stand. Tests showed that sensors placed in central part of catalytic bed where CO reheat and NO<sub>x</sub> reduction takes place undergo faster degradation. Beginning of degradation exhibits after 6 weeks of tests and results from separation of Pt electrodes and after-heater layer.

Destroyed sensors were examined by EDS, TEM and AFM methods. This allowed for identification of intermetallic compounds, PtSn<sub>2</sub> and PtSn<sub>4</sub>, which are responsible for lower adhesion of electrodes.

## Sensors for scanning probe microscopy (SPM)

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The characterisation of material properties in molecular or atomic dimensions together with a time resolution in the range of some ten femtoseconds is of increasing importance in the field of materials used in mechanical engineering, microelectronics as well as chemistry and biology. An outstanding solution of these problems up to date is the scanning probe microscopy (SPM) technique. But this is only true, if it is possible to design and fabricate appropriate probes adapted to the particular problem. For the mass fabrication of probes with reproducible properties the methods of microstructuring technologies including lithography together with wet and dry etching are applied which are already be known to be adequate from microelectronics and MEMS. Different probes optimised not only to measure topography but also other physical surface properties will be presented and discussed in some detail.

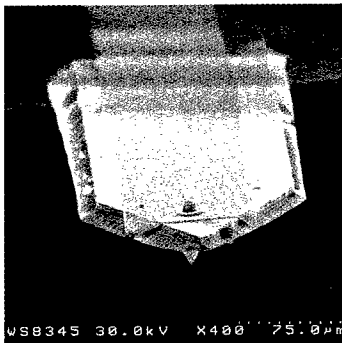


Figure 1: GaAs-Cantilever with an integrated VCSEL diode for SNOM.

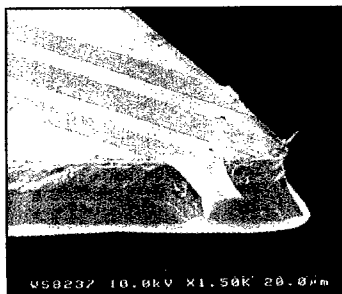


Figure 2: A coplanar waveguide GaAs cantilever with a FIB grown tip for USPM applications.

Fig.1 gives an example of a GaAs cantilever with an integrated tip in which a vertical cavity surface emitting laser diode, a VCSEL, is integrated. The laser light coupled into the metal coated tip from the rear allows to obtain a nanometer light source due to the miniaturized aperture at the tip apex. This probe is quite useful for a very compact set-up in scanning near-field optical microscopy (SNOM).

Fig. 2 shows another GaAs cantilever covered with a thin layer of LT-GaAs which carries a microwave coplanar waveguide. Together with a laser beam providing ultrafast optical pulses, a photoconductive switch is realised which allows to launch or detect pulses in the sub-picosecond range. This technique allows to combine both high lateral and temporal resolution in particular for voltage contrast measurements in ultrafast scanning probe microscopy (USPM).

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## Development of a high-k composite for integral capacitors and the electrical property

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Integral passives are being considered for use in multichip modules (MCMs) to reduce substrate size and manufacturing costs. Specifically, the development of an integral capacitor material and the processing method for MCM-L (printed circuit board: PCB) is becoming increasingly important. The dielectric material should have a high permittivity (greater than 150, according to NEMI roadmap) and be compatible with current PCB fabrication processes. A composite material consisting of an epoxy combined with a ferroelectric ceramic filler is a candidate for this application, since epoxies are well known materials in PCB fabrication. However, the permittivities that have been previously reported for these types of composite material are less than about 70. According to the Smith model for composite permittivity [1,2], the filler loading should be greater than 80%, which can be achieved only with a multi-modal size particle system. Particle packing calculations show that the diameter ratio of the smallest particle to that of the largest one should be less than 1/10, in order to load 80 vol.% ceramic particles [3]. We have investigated the effect of ceramic particle packing efficiency in the epoxy matrix on composite permittivity, using several kinds of particles of different size. We have also characterized the temperature dependence of the permittivity.

We examined bimodal and trimodal packing, using PMN-PT (lead magnesium niobate – lead titanate, Y5V183U; TAM ceramics; average particle size = 0.9  $\mu\text{m}$ ), BT-8 (pure  $\text{BaTiO}_3$ ; Cabot Inc.; average particle size = 0.15  $\mu\text{m}$ ), and BT-16 (pure  $\text{BaTiO}_3$ ; Cabot Inc.; average particle size = 0.065  $\mu\text{m}$ ). PMN-PT has a very high permittivity of ca. 18,000, although very fine particles (smaller than 0.1  $\mu\text{m}$ ) are not commercially available to date.  $\text{BaTiO}_3$  exhibits lower permittivity than that of PMN-PT, and very fine particles (smaller than 0.1  $\mu\text{m}$ ) are commercially available. The surfaces of all particles

were adequately treated with a surfactant to prevent agglomeration.

It was found that bimodal packing improves the composite permittivity and that the combination of PMN-PT with BT-16 yielded the best results (see Figure 1). The highest composite permittivity achieved using this packing concept was  $\epsilon_r > 90$  at 80 vol.%. The PMN-PT used in this investigation is classified as EIA Y5V category (operation temperature is between  $-30^\circ\text{C}$  and  $85^\circ\text{C}$ ). The temperature dependence on electrical

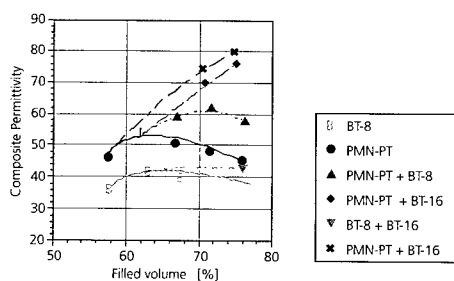


Figure 1: Effect of multi-ceramic epoxy filled composite system

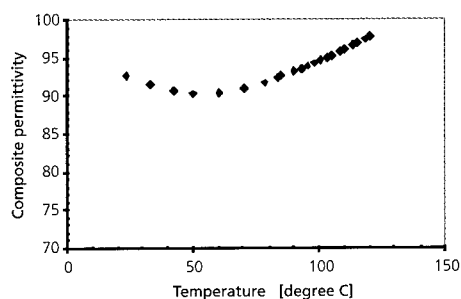


Figure 2: Temperature dependence of the permittivity for PMN-PT/BT-16 composite.

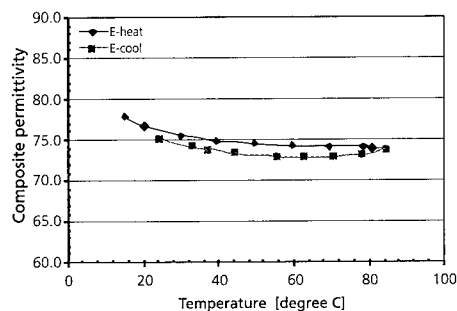


Figure 3: Thermal hysteresis of the permittivity for PMN-PT/BT-16 composite.

properties of BT-8 and BT-16 are unknown. However, they do not appear to be usable up to 125 °C (classified as category »7«), since BaTiO<sub>3</sub> has a Curie temperature at about 120 °C [4].

The temperature characteristics of composites would become a concern, when they are used as capacitors in electronic devices and operated under certain temperature range. We have investigated the temperature dependence of composite permittivity in the temperature range from 20 °C and 130 °C, and found that:

- 1 the composites retain the temperature characteristics of the ceramics used,
- 2 the Curie temperature of BT-16 is suspected to lie in the broad temperature range starting at > 80 °C, which would limit the higher operation temperature of PMN-PT/BT-16 composite to 80 °C.

Ceramic particles that have higher Curie temperature and are finer than 0.1 µm would be necessary for this type of composite material to be used as an integral capacitor.

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## Conducting atomic force microscopy – a new tool for the quantitative electrical characterization of thin oxides at a nanometer scale length

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The reliability of thin gate oxides and EEPROM tunneling oxides is of paramount importance for MOS integrated circuits [1]. A structurally and electrically homogeneous oxide is indispensable for device reliability, otherwise local weak spots trigger degradation and breakdown leading to early device failure [2]. Decreasing device dimensions and oxide thicknesses, the introduction of new dielectrics, like ultrathin oxides ( $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{Si}_3\text{N}_4$ ...); high epsilon and ferroelectric dielectrics, and the increase of process complexity intensify this problem [3], [4]. Conventional methods for oxide characterization, like electrical measurements on macroscopic test structures, transmission electron microscopy and emission microscopy lack either on lateral resolution or on electrical information and are no more sufficient for the characterization of today's and future dielectrics in microelectronics [5]. Instead, a technique is required, which allows the assessment of electrical and structural information with high sensitivity and lateral resolution.

In this paper, we present a new characterization method – Conducting Atomic Force Microscopy (C-AFM) – which we developed for that reason. The method is based on an AFM working in contact mode, which is equipped with a conductive tip and a sensitive current amplifier (RMS noise of 12 fA at a bandwidth of 400 Hz) [6], [7]. The setup allows the measurement of maps of local Fowler-Nordheim tunneling current through thin dielectrics simultaneously with the oxide topography and independent of it. In addition, local I-V curves can be obtained, which can be fitted to the well known Fowler-Nordheim tunneling equation to give the local electrical oxide thickness with an absolute accuracy of  $\pm 0.3$  nm and a relative accuracy of 0.1 nm [8].

The tunneling current images, obtained at constant applied tip sample voltage can also be converted to images of local electrical oxide thickness [9]. The lateral resolution of the method is below 10 nm for oxides with thicknesses smaller 20 nm and the oxide thickness sensitivity is 1 Angstrom.

Measurements on gate and tunneling oxides ( $\text{SiO}_2$ ) of different thickness are presented. They demonstrate the power of C-AFM for the investigation and analysis of thin oxides for R&D applications as well as for failure analysis. Further examples show results obtained on ultrathin oxides and ferroelectric dielectrics.

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## Laser beam micro joining for electronics industry

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The increasing packing density in electronics industry in the last years was the starting point to think about novel joining technologies. In the near future conventional processes will reach their limits and are going to be substituted by laser beam joining technologies. In particular for assembly, packaging and joining in chip manufacturing processes laser beam joining with high accuracy and the possibility of online process control will be introduced soon.

The conventional packaging processes like reflow soldering are not suitable for temperature sensitive components. The laser beam offers a very localised heat input due to the focus diameters below 200  $\mu\text{m}$ . The new diode lasers with increased beam quality and the modern fiber lasers are capable to join microparts with very low thermal load. High power diode lasers (HDL) are now used for joining processes like soldering and welding. For chip and wire connections high power diode laser beam soldering is a powerful tool to join the parts without mechanical deformation or ablation of material. Here copper wires have to be joined to copper pads on a silicon substrate. The dimensions of the joining zone are in the range of 100 to 200  $\mu\text{m}$ . The solder spheres (Sn63Pb37) with a diameter of 30  $\mu\text{m}$  are molten by a laser beam at 183°C. The problems of positioning the laser beam can be overcome by an integrated vision system. Possible solutions will be presented in the paper.

To demonstrate the capability of laser beam joining processes some applications of the different laser sources will be presented. They can either be classified by the wavelength

- High Power Diode Laser @ 805/940nm
- Nd:YAG Laser @ 1064nm
- Fiber Laser @ 1110 nm

or by the dimensions of the interconnection zone ranging from several microns for welding with Fiber Lasers to 4 mm for soldering with HDL.

The highlights of laser beam are

- very good focusability of the beam and therefore high intensities up to  $10^7 \text{ W/cm}^2$
- locally restricted energy deposition.
- optimal adaptation to the material by choice of the wavelength
- temporal and spatial controllability of the laser

The recent developments of new laser beam sources based on high power diode lasers and diode pumped solid state lasers enhance these qualities and open the field for further applications in microtechnology.

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## Strain fields associated with cracks in thin hard layers: an experimental study at different lengthscales

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We will present (preliminary) results of experiments designed to explore the limits of spatial resolution in the measurement of inhomogeneous strainfields that can be obtained with digital image correlation techniques. As a model system we have chosen the strainfields around crack tips in cracking thin hard layers. In particular the layers studied are ceramic (eg. TiC, TiNC) and are deposited by unbalanced magnetron sputtering on steel and aluminium substrates. Crack formation and propagation in such layers is of interest in its own right. It is of considerable practical importance because of the use as protective overcoats that these or similar layers find in an ever increasing array of applications. From a materials science point of view the fact that the interaction of the thin hard layer with the supporting metal leads to the formation of an array of similar cracks that propagate slowly is interesting. It opens the possibility to study crack initiation, propagation and interaction in detail.

We have set out to study the accompanying strain fields of such cracks with digital image correlation techniques, at very different experimental length scales. To this effect we perform *in situ* tensile loading in an optical microscope, an XL30 ESEM-FEG and a DI3100 SPM system. A wide range of magnifications and contrast mechanisms is therefore available. Images obtained at successive stages of deformation are correlated to yield strain fields under these varying circumstances, and the results are compared. As the events occurring at the crack tip are expected to be extremely localised the strainfields present are expected to show detail at all available magnifications. Therefore the results presented will be a good indication of the limits of spatial resolution in the determination of any inhomogeneous strainfield attainable with digital image correlation techniques.

## Properties of nanocrystalline diamond films deposited in argon microwave plasmas

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Nanocrystalline diamond films are recent materials with smooth surfaces, extreme hardness, low wear, low friction and good conductivity. They are suitable for several applications like tool inserts, micro-electromechanical-systems, biological sensors, medical implants, and surface acoustic devices.

A scanning acoustic microscope (SAM) has been used to characterize the propagation of surface acoustic waves of nanocrystalline diamond films. First measurements in the  $V(z)$ -mode using different ultrasonic frequencies detect a transversal acoustic velocity similar that of single crystalline diamond. These results lead to another measurement method in an image mode. The acoustic lens will be carried in a defined angular position. A surface image of the sample in the size of 1 mm x 1 mm is scanned. The analysis of the interference pattern gives the information of the exact transversal acoustic velocity and leads to the mechanical properties, e.g. Young's modulus  $E$  and Shear modulus  $G$ , of the investigated nanocrystalline films.

Complemental measurements regarding to the mechanical properties of these nanocrystalline films are micro-indentation measurements. They reveal superhardness and full elasticity. High resolution transmission electron microscopy gives an overview of the nanostructure of the investigated films.

The results received with the above described methods are compared with the known parameters of single crystalline diamond and lead to similar behaviour of these films. This fact opens a wide range of several industrial applications for the investigated nanocrystalline diamond films.

**Keywords:** nanocrystalline diamond, scanning acoustic microscopy, surface acoustic wave, Young's modulus

## State-of-the-Art micro materials models in MINIMOS-NT

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Considerable effort was spent on our two-dimensional device simulator MINIMOS-NT to get it ready for simulation of devices with high complexity and specificity in respect to materials, geometries, etc. Many of the existing physical models (band gap, mobility,

thermal conductivity, energy relaxation times, specific heat, etc.) were refined, some of them were replaced by promising new ones, and many new models were added as well. Being an ancestor of the well-known MOS device simulator MINIMOS [1], its experience with Si devices was inherited. Thereby, MINIMOS-NT became a generic device simulator accounting for a variety of micro-materials, including IV group semiconductors, III-V compound semiconductors and their alloys, and non-ideal dielectrics.

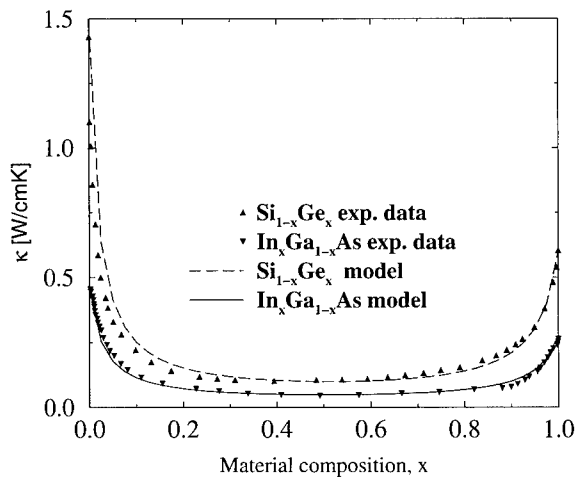


Figure 1: Material composition dependence of the thermal conductivity at 300K.

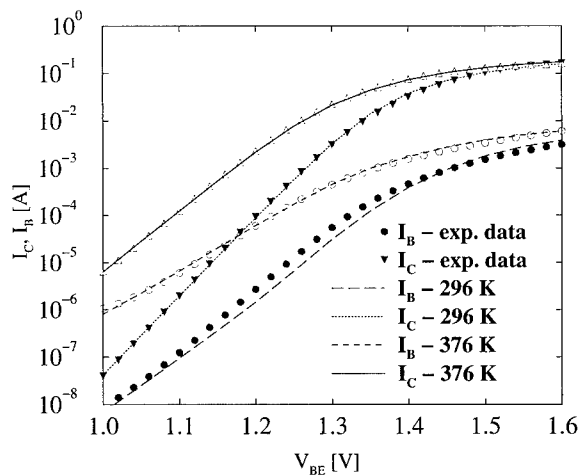


Figure 2: Forward Gummel plots at VCB = 0 V. Comparison with measurements at 296 K and 376 K.

For example, in Fig. 1 we present our model for the thermal conductivity  $\kappa_L$  applicable to all relevant diamond and zinc-blende structure semiconductors. The temperature dependence of  $\kappa_L$  is modeled by a simple power law which gives a good agreement with experimental data [2]. In the case of alloy materials  $A_{1-x}B_x$  it varies between the values of the basic materials (A and B). The model is used for device simulation with self-heating by solving the lattice heat ow equation self-consistently with the energy transport equations (system of six partial differential equations).

Several applications of industrial interest employ devices operating in a wide temperature range. Therefore, our models have been designed to meet this challenge in addition to the conventional Si applications. MINIMOS-NT has been successfully used for simulation of heterostructure devices, e.g. High Electron Mobility Transistors (HEMTs) and Heterojunction Bipolar Transistors (HBTs) [3]. For example, in Fig. 2 we present the simulated forward Gummel plot for an AlGaAs/GaAs HBT compared to experimental data. The simulation results at 376 K demonstrate the ability of MINIMOS-NT to reproduce correctly the thermal device behavior.

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## FEA modeling of FCOB assembly solder joint reliability

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The advancement in electronic packaging technology would enable greater applications for flip-chip-on-board (FCOB) assemblies. Eutectic solder bumps in flip chip are very much smaller than ball grid array solder joints and hence the thermal fatigue strength of the solder bumps becomes a major concern even with underfill encapsulation. Accelerated Thermal Cycling (ATC) and Thermal Shock (TS) tests of FCOB assemblies are used to assess the reliability of flip-chip solder interconnections. Non-linear Finite Element Analysis (FEA) is also widely employed to simulate the FCOB assemblies subjected to such reliability tests[1]. It is noted that some studies do not take into account the temperature ramp-rate or strain-rate effects in the solder properties and the cyclic frequency effects in the fatigue life prediction model. In this paper, non-linear finite element analysis modeling and simulation of ATC and TS reliability tests were conducted using eutectic solder properties which are dependent on both temperature and strain rate. Newly developed temperature and strain-rate dependent material properties for eutectic solder[2] was used to model the ramp-rate effects. New frequency modified Low Cycle Fatigue life prediction relationships[3,4] have been developed and used in this study. Both the plastic strain range and modified energy based driving force parameters were employed for predicting the solder joint fatigue lives for Accelerated Thermal Cycling(ATC) and Thermal Shock(TS) reliability tests. The FEA and life prediction results were compared to reliability test results for FCOB assemblies. It is interesting to note that the predicted results were able to predict the correct trend in the experimental results where shorter life cycles were noted from ATC tests and longer life cycles were recorded for TS test conditions. The predicted fatigue lives of the flip-chip solder joints gave good agreement with the experimental life cycles to failure measured by the Weibull mean-time-to-failure (MTTF) results.

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## Determination of heat treatment states fit for microcutting of steels

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Microcutting of microcomponents made of steels which have to bear high loadings imposes severe problems concerning the tools and the materials states of the workpieces. Here normalized materials states, as usually used in machining of macrocomponents, are not adequate because they need to be heat treated afterwards in order to adjust the properties necessary during service. They may cause distortions and therefore microcomponents which have to bear high loadings and should be produced by cutting, have to be heat treated before microcutting. Additionally, normalized materials states cause severe difficulties at microcutting. This is due to the fact, that ferritic and pearlitic regions with very different hardness values have dimensions of the magnitude of relevant tool sizes and therefore cause problems in the constancy of the machining conditions as the change of microstructure is noticeable.

This means that microcutting of steels has to be performed with heat treatment states which have finest and equidistantly distributed carbides and therefore show constant machining conditions on the scale of the tool size. This may be achieved by quenching and subsequent tempering, which leads to high strength states. As these may be difficult to machine, it is expected that at intermediate temperatures an optimum of machinability and service properties can be found. The investigations shown in this paper deal with the determination of this optimum at the steel SAE 1045 (German grade Ck 45), in which grooves were micromachined by milling after different heat treatments. For each of them, the cutting velocity, the feed per tooth and the depth of cut is varied and an ideal process parameter combination was identified. Thereby a close relationship between the material state and the process parameters was found. The materials states close to the surface produced by these processes were characterized using different techniques as light and electron microscopy, force-indentation-measurements and optical topography measurements. This allows the determination of heat treatment states of steels which are suited adequately for microcutting.

## The influence of surface oxide films on hardness and thermosonic wire bonding of Al bondpads

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The thermosonic (TS) Au/Al wire bonding process is influenced by a variety of very different factors, including the bonder setting parameters, the wire quality and the different material properties of the whole bondpad layer system. From a materials science point of view, the bonding process is based on a direct metallic contact between the Au wire and the Al metallization. Therefore, TS bonding requires a removal of the native Al oxide film formed in ambient air at the bondpad surface which is normally achieved by friction forces during the initial bonding contact. However, technological steps previous to wire bonding, like plasma etching or chemical cleaning may result in the formation of Al oxide films with increased thickness. Due to the high hardness and strength of Al oxide, the removal of these films during the bonding contact is much more difficult resulting in yield and reliability problems in wire bonding. Thus, techniques which can be applied to control the Al oxide formation at bondpads surfaces before bonding are of considerable practical significance.

The aim of the paper is to discuss the potential of nanoindentation testing methods for the detection of an excessive oxide formation at Al bondpads. Using an oxygen plasma treatment, the oxide film thickness was systematically varied between 5 nm (native oxide) up to values of 100 nm. The microstructure and thickness of the films was investigated by Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM) in combination with x-ray analysis (EDX-) techniques while the surface roughness was measured by Scanning Force Microscopy (AFM).

The results of indentation testing revealed a distinct correlation between the oxide film thickness and the observed hardness values. Accompanying numerical simulations using Finite Element Models (FEM) allowed to investigate separately the influence of different influencing factors on the indentation behaviour in addition to oxide film thickness, like Al yield stress, thickness of the Al layer or indenter tip shape. Based on these results, the intensity of oxide formation on the bondpads could be characterised by an appropriate analysis of the indentation experiments. In addition, wire bonding tests were performed for the same samples to characterise the influence of the different oxide films on the bonding quality. Series of bond loops have been realized and tested by Pull- and Sheartest methods. The results are correlations between Al-pad structure (mainly oxide configuration) and impact of the bonding parameters.

## Materials for molding processes in microsystem technology

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Microsystem Technology is predicted to become one of the most important technologies in the 21st century. Although the required materials properties are mainly determined by the demands of the application, some specialities of micro products have to be considered. Additionally, the relevant materials have to be suitable for being processed by enhanced micro manufacturing methods which often lead to further demands on the materials profiles. As typical representatives of these manufacturing methods, replication processes as well as the applied materials will be described in the following.

Thermoplastic materials which shall be suitable for molding of microstructures with high aspect ratios at first have to be characterized by a low viscosity. Other necessary properties are a high mechanical, thermal and mostly chemical resistance. Because of the small volumes the material costs are less important compared to conventional injection molding, so that high performance polymers like PEEK or PSU are often applied. Some examples for injection molded microstructures will be shown.

Photocurable reactive resins are applied for an approach similar to rapid prototyping. Photoinduced molding of low viscous resins under ambient conditions leads to significantly reduced cycle times. Additionally rapid testing of new composite materials, e.g. polymers filled with titanium or zirconium oxide with particle dimensions in the nanometer range, is easily accessible. Microcomponents molded from polymers and different composites like dyes with nonlinear optical properties and nano sized ceramic powders will be presented.

Metal or ceramic materials are applied if polymers can not provide the required materials properties. For manufacturing metal or ceramic microstructures, the powder injection molding process including debinding and sintering has been developed according to the special requirements of micro parts. Sintered structures in different metals (e.g. 316L), hard metals and ceramics (e.g.  $\text{Al}_2\text{O}_3$ ,  $\text{ZrO}_2$ ) will be shown. Minimum structural details are actually in the range of 50 microns. For further reduction, application of powders with diameters in the submicron or even nanometer range is necessary.

## Modelling of thermal aspects of power electronic assemblies

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One of the main issues of electronic packaging is the removal of heat generated by power losses. This is of concern especially in the application of power electronics. Different models will be described which allow the assessment of individual phenomena of the heat transfer problem, i.e. heat spreading in layer structures, heat transfer by convection, or the transient thermal behaviour of assemblies. Here, emphasis is put on »simple« models, which are best suited to check for the main influencing parameters while keeping the modelling effort small. Comparison with experiment always has shown satisfying agreement.

The increase of the thermal resistance with life time affects the reliability of an assembly, e.g. by fatigue crack growth within the solder layer. It will be shown, that thermal modelling ahead of a life time experiment assures optimum setting of test parameters in order to minimise experimental effort. In addition, measured changes of the thermal behaviour can be used as a measure of damage once the tendencies have been verified by suitable thermal modelling.

## Paraelectric film as microwave phase shifter

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Beam scanning in the phased array antennas is produced by the many elements that emit signals with various phase shifting. As a result, the narrow microwave beam is formed and electronically steered by the manipulation of relative phasing of individual antenna elements which quantity might be  $10^2$ - $10^4$ . Previously, in the military applications, such antenna was a complex and expensive system with the limited and peculiar use. However, nowadays a rapidly developed telecommunication need small size cheap MMIC phased antennas to exchange information between several satellites, between satellite and base stations, and in the future even for any mobile for direct communication through the satellites.

The key antenna's component is phase shifter. Powerful military antennas usually are based on ferrite phase shifter (sometimes *PIN* diodes are used as well). Both of those types of components have some limitations, and phase shifter need to be improved to be applied in the microelectronics.

Very promising alternative for phase shifter components is to use a low loss *paraelectric material* which dielectric permittivity  $\epsilon$  could be fast controlled by the electric bias (controlling) field  $E$ . Advisable in this proposal paraelectric is responsible for  $\epsilon(Ea)$  fast control up to 50%. To decrease the controlling voltage, active paraelectric should be used in a form of thin film deposited on substrate. In separate phased antenna a high thermal transparent crystals ( $\text{BeO}$ ,  $\text{MgO}$ ,  $\text{Al}_2\text{O}_3$ ) might be used. The proposal is to use as a substrate crystals common in the MMIC: semi-insulating GaAs or other  $\text{AlIBV}$  crystals, as well as the high-resistance Si.

The effect of dielectric permittivity control at microwave as well as this effect application in a phase shifter has 40 years history. However, only now the processing of ferroelectric thin film becomes such a perfect that tuned microwave device based on paraelectric film can be competitive with the microwave ferrite and *PIN* diode. To compare with microwave ferrite devices ( $\mu$ -controlling) and semiconductors ( $\sigma$ -controlling), the advantages of  $\epsilon$ -controlling are the higher operation speed, increased microwave power, lower cost and size, as well as a possibility to use paraelectric device at much higher frequencies. Previously the only one paraelectric material was a candidate for microwave applications, namely the  $\text{BST} = (\text{Ba}, \text{Sr})\text{TiO}_3$  composition. Nevertheless, the other paraelectric solid solution:  $\text{PST} = (\text{Pb}, \text{Sr})\text{TiO}_3$  might have better performance, and perfect epitaxial films with  $\text{PbTiO}_3$  as a part of compositions are elaborated by the laser ablations on the  $\text{MgO}$  substrate.

The improvement of tuned microwave paraelectric device is expected with the use of multi-layered (heteroepitaxial) thin film structure only, in which one can operate by the  $\epsilon$  magnitude as well as  $\epsilon$ -change under the bias (controlling) field:  $\epsilon(E_b)$ . Paraelectric microwave phase shifter consists of five important components: paraelectric-like thin film, dielectric substrate, electrodes and interfaces film/electrode and film/substrate.

Heteroepitaxial (multi-layered) structure is quite favorable also to obtain desirable microwave performances (impedance, phase shift, insertion loss, etc.) by the optimization of metallic guides and dielectric layers collocations.

Nanoscale structure opens the possibility to modify paraelectric film properties: the  $\epsilon$ -magnitude and  $\epsilon(E_b)$  dependence, as well as microwave loss factor. Heteroepitaxy is used to control Curie temperature through the match or misfit of the adjoining layers lattice parameter. The compression or tension of the nanoscale layers strongly depends also from a matching in thermal expansion coefficients of substrate and multi-layered film. All mentioned properties should be simulated before the film deposition.

The design of phase shifter was elaborated with a use of simulation. Multi-layered structure is quite favorable to obtain need microwave performance at a given frequency range by the optimization of metallic guides and dielectric layers collocations and parameters. Phase shifter is elaborated for the frequency range of 10–40 GHz.

Input data for simulations were the film  $\epsilon$  and its change under the bias (controlling) field. These data were obtained experimentally from the slot (planar) capacitor investigation at 1–10 MHz, and this is possible because paraelectric has no dielectric dispersion up to 300 GHz. Appropriate microwave loss tangent was obtained from the bulk paraelectric materials microwave previous investigation, and the conductivity of suited metals was used in our simulations as well. Output data of calculations were need planar geometry of all materials collocation to satisfy standard  $\sim 50$  Ohm impedance and to obtain a maximum relation the phase shift to the insertion loss.

After the comparison of all used in the MMIC transmission lines, the coplanar line has been chosen as a basic line. While simulation of electromagnetic field in microstrip lines and slotlines, the 2-D problem was reduced to scalar partial differential equation of component Hertz's vector using generalized Neumann boundary conditions. This equation was solved by the finite elements method. Correspondent software for estimation of phase shifter characteristics was elaborated: the distribution of electromagnetic field in multi-layered structure, its wave impedance, wave-length, effective  $\epsilon$ , controlling phase shift and insertion loss. As a result, the clear recommendations were obtained as to the best collocation of film and metallic lines in dependence on applied materials parameters and geometry.



## FE-simulation of anisotropic damage development in creep process

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During loading processes, engineering materials undergo micro-structural changes. The irreversible micro-structure changing such as nucleation, growth and coalescence of microvoids and cracks, which is often observed typically in metallic alloys undergoing a creep deformation, are damage processes. Though damage evolution is a micro-process, its effects can be observed: decrease of YOUNG's modulus, increase of deformation rate and fracture of the engineering components. That means, the microscopic material damage is related naturally with the macroscopic behavior.

As damage develops generally anisotropically, it results in anisotropy. This work focuses on the simulation of the anisotropic development of the material damage and its influence on the creep deformation behavior. An anisotropic constitutive damage model is used for this purpose. In this model, a second-order symmetric tensor is used as the thermodynamical state variable for the description of the anisotropic material damage. The influence of the damage-induced anisotropy on the damage evolution is considered and the tensile stresses are assumed to be responsible for the damage development. This damage model is combined - according to the effective stress concept of Continuum Damage Mechanics - with the unified viscoplastic model of CHABOCHE by introducing an effective stress tensor. The resulting viscoplastic model coupled with damage is implemented into the Finite Element Program ABAQUS and is used for the FE calculation of the present work. In order to observe the anisotropic character of material damage easily, a square plate with a circular hole in the middle under various creep loading conditions is chosen as the main object for the simulation. Damage and deformation of the plate under these loading conditions are investigated and compared.

## An assessment of mechanical properties of mismatched weld joint with thin layers

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Some groups of weld joints are often highly inhomogeneous. Changes in the material structure are directly related to the mechanical properties in that area, e.g., tensile yield point  $R_e$  and  $R_m$ . Considering the above - mentioned problem of mis - match welded joints, it is essential that a mathematical model which shows the real condition of the joint is presented. The components of the state of stress at interfaces are determined what enables to establish the average value of stresses of the layer (W),  $\sigma_{ever}$ , at static tension as:

- undermatching case -  $(R_e^{W(un)}) < R_e^B$

$$\sigma_{ever}^{un} = K_W^{un} \cdot R_e^{W(un)} \quad (1)$$

- overmatching case  $(R_e^{W(ov)}) > R_e^B$

$$\sigma_{ever}^{ov} = K_W^{ov} \cdot R_e^{W(ov)} \quad (2)$$

where:  $K_W^{un}, K_W^{ov}$  - constraint factors of under- and overmatched weld joints.

The following revealed features of under- and overmatched weld joints were established:

- state of stress in heterogeneous weld joints under static tension [1];
- average strength  $\sigma_{ever}^{un} = f(R_e^{W(un)}, q, \kappa)$ ;  $\sigma_{ever}^{ov} = f(R_e^{W(ov)}, q, \kappa)$  of under- and overmatched weld joints,
- constraint factors  $K_W^{un}, K_W^{ov}$  which described change of mechanical properties of (W) zones as a result of stress state in that area,
- relative thickness  $\kappa_{cr} = f(K_s, \gamma^T, q)$  of the zone (W) which has no negative effect on the strength at static tension in undermatched weld joints as follow:

$$\kappa_{cr} = \frac{1-q}{2\sqrt{3}(1-q)K_s\gamma^T - \left[ \frac{\pi}{2} + 2(1-2q)\sqrt{q(1-q)} - \arcsin(2q-1) \right]} \quad (3)$$

$0 < q < 1$ ;  $K_s = R_e^B / R_e^{W(un)}$ ;  $\gamma^T = R_m^B / R_e^B$

- the condition expressed by parameter  $k$  when the mismatched weld joints fail in the ductile mode.

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## Large area 4H-SiC power Schottky diode

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### Introduction

SiC, especially 4H-polytype presents a great promise in the fields of high temperature, high power and high voltage devices due to its excellent physical and electronic properties. The wide band gap, high critical electric field strength, high thermal conductivity and high saturation velocity of carriers make it an excellent material from the point of view of high power devices for both low and high frequency applications. But the first step to make the device is to form the contacts to the SiC semiconductor structure. In our best knowledge no results have been reported on large area and acceptable thickness metal contacts to the 4H-SiC structures. In this paper, we report the initial attempt to form thick (about 100 nm) large area metal contacts to 4H-SiC substrate with epitaxial layer using diffusion welding technology.

### Method

Diffusion welding is a solid state joining process, which can be utilised to bond »difficult to join« materials. The 4H-SiC substrate was sectioned by quadrangles with 10-mm sides by the vendor (Cree Research Inc., USA). Prior the metallization the 4H-SiC quadrangles were given to chemical clean during 10 min. ( $\text{NH}_4\text{OH}:\text{H}_2\text{O}_2:\text{H}_2\text{O}::1:1:5$  solution followed by  $\text{HCl}:\text{H}_2\text{O}_2:\text{H}_2\text{O}::1:1:5$  solution at 70 °C). After cleaning the SiC was ultrasonically treated in acetone and ethanol. The Al foil was dipped for 1 min in solution consisting of  $\text{H}_3\text{PO}_4$ ,  $\text{CH}_3\text{COOH}$ ,  $\text{HNO}_3$  - 18 ml,  $\text{H}_2\text{O}$  at the temperature of the solution 70 °C. This treatment can leave a thin film of aluminium phosphate on the surface of Al and the foil was ultrasonically cleaned in 50% nitric acid to remove the film and then the Al was also ultrasonically cleaned in ethanol. Such scheme enabled to form all the contacts of the rectifying element of the modern semiconductor device simultaneously.

### Materials

In our experiments for the contact aluminium has been chosen for a contact material because of its low melting point (later in high power applications, other metals should be used due to the same reason), relative inertness to SiC in the solid state, and its importance as a matrix material in metal/semiconductor composites. For the process the 0.05 mm thick 99.99% Al foil with diameter 8.0-mm has been used. The SiC, used in our experiment was 4H-SiC substrate with the specification: diameter 35.0 mm, thickness 0.33 mm, conductivity - n-type, dopant nitrogen, net doping density range  $8.5 \times 10^{18} \text{ cm}^{-3}$  ( $N_D - N_A$ ), with 5 mm thick epilayer with concentration  $2 \times 10^{17} \text{ cm}^{-3}$  and surface treatment - carbon face polished as specified.

### Results

The vacuum diffusion welding process was realised in special equipment UDS-5 with various combinations of temperature and pressure. The duration of process was 500 sec. For determining the quality of the adhesion after bonding process the aluminium foil was pulled off the SiC surface. For the first approach pure qualitative examination was used. Table 1 shows the results.

Temperature [°C]	Pressure	Bond quality
500	20 - 50	None
550	20 - 50	Bad
600	20	Bad
600	30	Very good
600	50	Excellent

For the manufactured large area (over 50 mm<sup>2</sup>) Schottky diode examples the electrical measurements have been carried out. The U-I characteristics were measured and the temperature dependence of barrier height was defined in an analytical form.

Table 1

## Measurements of the Young's modulus of ceramic substrates in the range of 20 – 300°C

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The construction of new high performance microsystems which can show a high reliability in a wide range of temperature requires an assessment of the thermo-mechanical behaviour of the different combined materials. Than it is possible to evaluate the occurring of stress regions in the components and to calculate the life time of the microsystems. For modelling the thermomechanical properties by Finite Elemente Simulation the coefficient of the thermal expansion (CTE) and the Young Modulus as a function of temperature of the different materials has to be available.

The elastic stiffness of materials can be measured with high precision resonant beam techniques.

Taking into account the standard DIN V ENV 843-2 Young Modulus measurements of different ceramic and glass-ceramic composite packaging materials were carried out in the temperature range of 20 – 300 °C in vacuum by use of the measuring system Elastotron 2000 (HTM Reetz GmbH, Berlin). The elastic moduli are determined from the resonance frequencies of the flexural vibrations of a beam.

The measuring results for different high alumina ceramic tapes, glass-ceramic composite monolayer tapes showing different thickness and glass-ceramic composite multilayer LTCC (Low Temperature Cofiring Ceramics) prepared by use of different numbers of substrate layers are discussed.

## Materials science and engineering – A major topic in the future of microelectronic packaging

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The requirements of future products are the driving force for new packaging technologies. Microelectronic packaging is most important for the functionality, quality and economy of microelectronic products. It has to provide new solutions for a wide range of applications. The product range of advanced microelectronic systems can roughly be split into cost and performance sensitive portable systems, commodity products, automotive products and high performance systems. Examples are packaging technologies for ultra-thin systems (smart cards, memory modules), systems with the highest degree of miniaturization (smart watches, pagers), light weight and ergonomically correct communication interfaces (mobile phones, notebooks), high clock rate and low power systems (telecommunication, desktop PCs), high interconnects density – low-size – low-weight products (workstations, avionic systems), and environmental resistance – high operational temperature – low-size – low-weight – low-cost systems (engine controllers, ABS). To realize an optimized package (single-chip, multichip module) qualified materials selection, manufacturing processes and controls are required. It is necessary to coordinate the design with manufacturing and testing as well. Additionally, for all future microelectronics developments, environmentally compatible packaging technologies and materials should be applied. Due to the high circuit density and very localized high power dissipation, temperature rise is an important issue affecting the performance and reliability as well. New demands on the reliability of high temperature electronic and mechatronic systems have to be accepted. So, all new packaging architectures also require thermal and thermo-mechanical analyses to ensure the high level reliability goals. New testing techniques (e.g. the microDAC technique which can be used to get more detailed information on local stress and strain fields) and powerful simulation techniques have to be combined to optimize interconnection and packaging technologies based on the latest achievements in advanced materials science.

Today the main goal for all packaging requirements is cost reduction. Besides low-cost material selection, a significant decrease in cost can be achieved by developing new technologies with fewer processing steps. However, packaging also has to create an economic link between chips and products. Therefore, a further driving force for new packaging technologies is given by the trend of chip characteristics. The proposed 300 mm wafer technologies will offer complex chips; they will stimulate a new area of microelectronic products and thus appropriate packaging technologies, too.

Future packaging requirements will be defined by products especially designed to suit the needs of the globally acting individual, who on every place on earth has access to communicate and universal individual access to information. To meet these challenges, powerful portable mobile products need to be developed. This can be achieved through

small, light-weight and foldable products. Since they are to be carried on different parts of the body, they should be interlinked in order to increase their efficiency. A long operating period requires efficient generation and storage of energy, as well as extremely low energy consumption. The information exchange with the office is carried out using a portable »Electronic Scout«, for example in the form of a »Smart Foil«. At the office an »Electronic Secretary« could be available to coordinate all the incoming information. The portable »Smart Foil« must be small, light, and foldable, and yet have written, oral and visual information in- and output. It allows wireless communication with data networks and is its own energy provider. It helps create texts, diagrams and pictures. In combination with a »Smart Watch« or a »Smart Shoe« this »Electronic Scout« will serve as Handy, Pager, PDA etc. The »Smart Shoe« will take over the task of the worldwide data transmission since in this case energetic problems can be easily solved. The »Smart Foil« then functions as electronic newspaper, navigation system and travel guide. The medical monitoring of the globally active individual is taken over by an intelligent health data station, which via the »Smart Shoe« communicates with the »Electronic Secretary«, which in turn organizes medical help if necessary. All necessary information, e.g. addresses of doctors, health status etc. may be recorded in the »Electronic Secretary« and can be easily accessed. The requirements for the realization of a mobile management system are thus fulfilled. The realization of these visions puts high demands on the development in different fields: materials research, data processing, energy supply and system integration (Packaging & Interconnection).



## Correlation of reliability with the mechanical properties of leadfree solders

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The DGX I proposal suggests to prohibit the use of lead in electronic assemblies in 2004, making the employment of alternative lead-free solders necessary. The aim of this work is to determine the manufacturability and reliability of lead-free solder for electronic applications and to investigate the fundamental material properties influencing the reliability. The following alloys have been selected based upon typical solder-requirements, such as price, availability, melting range, wettability, creep, yield strength and Young's modulus: BiSn42 (139 °C), SnAg3.8Cu0.7 (218 °C), SnAg3.5 (221 °C), SnAg3.5Bi4 (213 °C) and SnCu2Sb0.8Ag0.2 (218 °C-229 °C), with SnPb36Ag2 (179 °C) used as the reference material.

SMT-assemblies were built up on FR4-substrates with bare Cu. The leaded and unleaded components had finishes containing lead as well as being lead-free. In an initial test the minimum soldering temperature was determined. Afterwards the assemblies were exposed to both thermal cycles from -40°C to 125°C and stored at 125 °C for 1500h.

First of all the manufacturability and the results of the reliability up to 3000 thermal cycles for the various solders with different SMDs will be shown. Further information on changes in the microstructure, the affinity to form surface cracks and the crack growth will be presented. Future possibilities to increase the reliability of lead-free solders will be discussed.

## Use of alternative materials for packaging of sensors and MCMs

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One of the major disadvantages of present day's plastic packages made of thermoset molding compounds is their limited suitability for operation at higher temperatures. Also the full encapsulation process of the die and the interconnections is a source of potential failures. Furthermore due to the constituents which make it flame retardant the material of the standard housings is not desirable under environmental aspects.

It was the aim of the work to explore the possibilities of alternative packaging materials and the development of a new type of low cost plastic package for sensors and MCMs. Hence the so-called premolded packaging technology using high-temperature thermoplastic materials was selected. The process comprises several individual steps. In a first step the premolded parts, i.e. the body of the package incorporating the leadframe and possibly a leadframe as well as the lid are injection molded. Specific details, advantages and possibilities of the injection molding are demonstrated.

After further assembly and interconnection bonding of the electronic device the parts are joined by welding. This step is a very crucial one as it determines the hermeticity of the package. Different welding techniques like ultrasonic welding, orbital welding and laser welding have been evaluated. The design variations and results of the welding optimization are shown in the case of a PLCC and a QfP package. Furthermore the paper demonstrates design optimization and selected properties of these packages.

Finally the premolded packaging technology is compared to the standard molding technology taking into account aspects like manufacturability, performance and costs.

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## Interconnection materials for flip chip technology in high temperature automotive applications – a comparative study

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The automotive electronic industry is facing a trend towards increasing functionality of electronic control units (ECUs) and the demand for smaller ECU size. This asks for new interconnection technologies such as flip chip technology. Today flip chip technology is typically applied to products with modest ambient temperatures (up to 85°C) and reliability tests are usually performed at temperatures below 125°C. Automotive applications however are subjected to harsh environment with high temperatures (up to 150°C), extended temperature cycles and strong vibrations.

In this paper we present a comparative study of interconnection materials for flip chip technology in a high temperature environment where junction temperatures of 150°C and more are possible. Test samples were built on a ceramic substrate realizing the interconnection with various solder materials as well as conductive adhesive. After applying and curing the underfill the samples were subjected to lifetime tests (vibration, temperature storage and temperature cycling). Up to now the solder joints passed all tests for typical automotive requirements. Technological limits are under investigation. Adhesive joints showed comparable reliability, but also higher process risks. Additionally, adhesive joining causes design drawbacks (lower thermal conductivity and higher impedance).

## Interface design and thermal stresses in layered microelectronic assemblies

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In microelectronic components thermal stresses arise from the thermal and elastic mismatch of the bonded materials. With increased requirements in the reliability of microelectronic components and packages sophisticated analysis methods including interface behavior become necessary. The incorporation of continuum mechanics descriptions of interfaces into the finite element method and the application of this approach is demonstrated here.

The present paper uses a fracture process zone (FPZ) model to describe the non-linear behavior of interfaces. A cohesive strength and a cohesive energy characterize the interface with both material parameters being embedded in a traction-separation law.

With the FPZ model cracking in bi-material strips under thermal loading is studied. Initiation and propagation of cracks is predicted in dependence of the applied temperature change. Cracking occurs at a critical temperature which depends not only on the property mismatch of the two solids but even stronger on the properties of the interface. Subsequent material separation is dominantly in shear. The crack growth rate is found to be very large initially but decreases to zero as the crack propagates. Based on these simulations the model shows a path towards increasing robustness in electronic packing by appropriate interface design.

## New lamination technique to join ceramic green tapes in multi-layer processing

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Lamination of stacked ceramic green tapes is used on a large scale in the manufacturing of capacitors, packages, multilayer circuits, actuators, and gas sensors. Lamination produces homogeneous junctions between green tapes, in which after sintering the original interfaces cannot be detected anymore. The quality of the laminated interface, and its stability during the binder burnout and sintering process strongly determines the quality of the final product. Partial metallization of the tapes complicates this process. A brief overview of today's lamination techniques concerning quality issues and limitations of these processes will be given.

During the common thermo-compression method, the binder phases of both green tapes are joined together at elevated temperatures and pressures. Typical temperatures and pressures are 50 to 80 °C and 3 to 30 MPa. During thermo-compression, a thorough interpenetration of the powder particles in both unsintered tapes must be achieved. This requires specific green tape microstructures with regard to the ratio of powder, binder, and porosity. Because of mass flow, which is caused by the applied pressure and temperature, the method cannot be used to produce complex 3D multilayer structures or fine patterns for micro systems.

A new lamination technique will be presented which can be performed at low pressures and room temperature. In contrast to the thermo-compression method, the new cold low pressure lamination technique reduces deformations, and will be important in the preparation of laminated fine, undercut, complex three-dimensional structures. The method is based on a gluing step. The two green tapes are stuck together by an adhesive tape at room temperature under very low pressures. Even though the tapes are separated by the adhesive tape and no interpenetration of the particles can occur, the tapes are joined after sintering. The joining mechanism will be described and advantages of this new technique will be addressed.

## Reliability investigations of vibration excited circuit boards

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Circuit boards as parts of electronic equipment mounted in space shuttles, in aircrafts, in cars or in mobile measuring systems must possess a high reliability. This fact is especially important if the circuit board has a relevance for system's safety. During the design process the reliability can be measured and estimated. This estimation is based on numerical simulation. For the simulation input values are necessary. Such input values for reliability investigations which can be experimentally determined are vibration amplitudes or eigenfrequencies.

The paper describes an evaluation framework of circuit board reliability based on measured eigenfrequencies and amplitudes. The valuation includes the following main steps:

### 1 Experimental vibration investigations of the board by means of a laser scanning vibrometer

By a special structure borne noise excitation in a definite frequency range the vibration behaviour of a mobile measuring system's circuit board is investigated. Electronic components on this board with high amplitudes have to be detected.

### 2 Microscopic analysis of solder joints

A metallographic analysis of solder joints of components with a high amplitude is performed to get exact geometry data for stress calculation.

### 3 Stress calculation of solder joints

A local 3-dimensional FE-model based on the experimental results is created. Calculation of stress under test conditions and valuation in comparison with critical stress values.

### 4 Estimation of solder joint damage

A so called »Cumulative Damage Index« which is a measure for a failure-probability has to be calculated. Is the index lower than 1 no solder joint damage has to be expected.

The vibration investigation of a first example showed a high load at a board-mounted plug. The amplitude of the board rigid body mode (10 Hz) and so the amplitude of the plug is depending on measuring point between 105 and 140  $\mu\text{m}$ . In the first board mode shape (64,4 Hz) the highest amplitude of the plug was 6  $\mu\text{m}$ . This value consists

of a rigid body and of a bending share. The rigid body share was bigger than the bending share. The bending share is 1,2  $\mu\text{m}$ . These values (10 Hz - 140  $\mu\text{m}$  and 64,4 Hz - 1,2  $\mu\text{m}$ ) used as input values for the numerical simulation.

The calculated "Cumulative Damage Index" values are  $10^{-22}$  (10 Hz) and  $10^{-2}$  (64,4 Hz). This means that no solder joint damage due to vibrational fatigue has to be expected.

A same valuation was done with a second circuit board. Here a PQFP (Plastic Quad Flat Package) was identified as a critical component.

## Mode shape analysis of microstructures by means of laser-optical methods

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For two types of microstructures mode shape analyses are necessary:

- for sensors and actuators which have to vibrate themselves e.g. micro mirrors and
- for microstructures which have to work in a vibrating environment e.g. sensors in aircrafts, helicopters or in automotive applications.

Microactuators as well as microstructures used in vibrating environment have to possess a high reliability. To realize carefully directed solutions the development engineer usually needs the knowledge of the dynamical behaviour of such systems, that means eigenfrequencies and mode shapes should be known. For their contactless determination different laser optical methods are known:

- Holographic Interferometry,
- Single-point vibrometry coupled with a modal-analysis system,
- Scanning vibrometry and
- Auto-focus method.

The methods can be used for investigations under operating conditions (micro mirrors) or analyses under separate vibrational excitations e.g. with a shaker (sensors in a vibrating environment).

The poster describes the coupling of a single-point laser vibrometer with a modal-analysis system exemplarily shown by vibration investigations of micro mirrors. The development of a test stand is explained. Important results (frequency response functions with eigenfrequencies and mode shapes) under operating conditions and under separate excitation by shaker are described. Frequency response functions have been measured at points of the mirror, the housing and the test stand. Relevant eigenfrequencies have been detected at 1504, 1711, 1988 and 2827 Hz. Between 5160 and 5784 Hz eigenfrequencies of the housing which excite also the mirror were measured.

Measurements performed with a laser scanning vibrometer are shown, too. This faster working method allows to investigate more complex structures in shorter time. Results of laseroptical modal analysis could serve as foundation for numerical simulations.



## Distributed crack propagation in porous ceramic materials

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Constitutive modelling of polycrystalline porous ceramic materials obey description of the:

- elastic deformations of initially porous material,
- shear bands dislocation existence and
- deformations connected with cracks propagation.

The proposed general constitutive equation for quasi-brittle material will contain two internal parameters: porosity parameter and second order damage tensor. Thus the compliance tensor describing material response is a function of these internal parameters.

The initial porosity of the material is assumed to be closed and distributed in grains and along grain boundaries. Both types of porosity influence the initial value of the compliance tensor and the initial stage of deformation process, i.e. elastic deformation. We assume that porous shapes are spherical or ellipsoidal and their distribution inside Representative Surface Element (RSE) is initially homogeneous.

When the state of stress increases the shear dislocation bands are created in a certain part of grains in RSE. The plastic deformation describing this phenomenon were included to description of the material response by application of the Eshelby inclusion model.

The third part of deformation process is connected with crack initiation and kinking along grain boundaries. The description of crack propagation process is strongly influenced by grain boundary porosity. Crack can propagate if the energy release rate criterion is satisfied. The estimation of deformation of growing cracks inside the polycrystalline material is performed by averaging procedure over RSE.

A numerical example of the quasi-static loading process of MgO polycrystalline ceramics is analysed as a two-dimensional continuum containing defects.

## Interface studies between epoxy and recycled rubber

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This work covers studies on interfaces between epoxy and recycled rubber used as toughening phase in epoxy resins. Different surface modification techniques were used to improve the compatibility at the interface between the rubber particles and the epoxy matrix. Silane coupling agents, plasma surface modification and acrylic acid/benzoylperoxide were used to enhance the surface properties of rubber particles. Grinded rubber particles with or without surface treatment, were mixed with epoxy. This mixture was poured into molds to obtain mechanical test specimens. After curing, mechanical tests were performed and the fractured surfaces of the specimens were examined under Scanning Electron Microscope, SEM.

Surface treatment of rubber resulted in better mechanical properties as compared to the samples prepared with untreated rubber due to better adhesion created at interfaces. On the contrary to the tension and impact test results, fracture toughness values showed better improvements when epoxy was modified by surface treated rubber. The main rubber toughening mechanisms observed by the use of SEM studies were »shear deformation« and some »debonding« initiated at the interfaces.

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## Development of in-situ nanoindentation system in a scanning electron microscopy

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Depth-sensing indentation test is a prominent method of measuring mechanical properties of micromaterials. The developed system is characterized by its small sized testing machine and by its operation in a chamber of a Scanning Electron Microscopy. Since, the system is useful for direct observation of local material behavior during nanoindentation in a microscopic field of view and for in-situ testing of mechanical behavior of micromaterials or microstructures. Specimen is subjected to an X-Y stage driven by ultrasonic linear motors. The indenter is installed at the end of a load cell's cantilever of 10 mm in length and the load cell detects the indentation load. The load cell is installed at the center of the beam as shown in Fig.1. The indentation to a test material is accomplished by piezoelectric linear actuators that are installed at both ends of the beam. The displacement of the beam (load cell)  $\delta b$  is detected by the differential transformers. The actual displacement of the indenter is determined by the consideration of  $\delta b$ ,  $\delta c$  and  $\delta s$ . Here,  $\delta c$  is a deflection of the cantilever of the load cell during indentation and  $\delta s$  is a deflection due to the stiffness of the system such as X-Y table. This system enables us to analyze local materials behavior on the basis of the microstructures such as grain boundaries or dual phase structures and contributes to establish the evaluation method of micromaterials in a micro/nano-scopic scale.

## Micro/nano formability of amorphous alloys in the supercooled liquid state

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There are two major requirements of micromaterials for fabrication of microcomponents and microsystems. First, material characteristics must be exploitable on a microscopic scale in addition to conventional macroscopic scales. Second, forming, processing and machining methods to form micro geometries must be established. Amorphous alloys are highly useful for realizing high-performance micro actuators and structures due to their excellent characteristics as functional or structural materials, including isotropic homogeneity free from crystalline anisotropy. The present report introduces new materials which are second-generation amorphous alloys and their microforming properties.

If an amorphous alloy is heated again, the alloy usually crystallizes at temperature  $T_x$ . However new amorphous alloys shown in this report reveal obvious glass transition behavior at temperature  $T_g$  and develop the supercooled liquid state in a wide temperature range. In the supercooled liquid state, the materials reveal perfect Newtonian viscous flow characteristics and have a great advantage in achieving deformation under very low stresses compared to conventional plastic deformation, furthermore, exhibit an excellent property of microformability on a submicron scale.

To evaluate the microformability of the materials, we developed an evaluation system using a micro V-grooved die made of (100) silicon that has been processed by electron beam lithography and anisotropic etching to quantitatively evaluate the microscopic formability for the materials. The V-groove is 0.2 to 20 micrometers wide and has a base angle of 70.6 degrees. The materials are La-, Zn- and Pd-based amorphous alloys and are subjected to microforging and die forged with the micro V-grooved die. After the deformation, we measured the shape of the specimen by using an Atomic-Force Microscopy. From these geometrical analyses of deformed specimens, we obtained the evaluation index as a percentage of flow area  $R_f = A_f / A_v$  ( $A_f$ : inflow area of specimen into the V-groove,  $A_v$ : V-grooved area of the die) and curvature  $1/r$  ( $r$ : radius of the tip of the deformed specimen). Excellent microformability should be obtained for the amorphous alloys because it has no anisotropy in crystalline structure and it is homogeneous with no grain boundaries or segregation.

As a result, amorphous alloys in supercooled liquid state exhibit Newtonian viscous flow and an excellent microformability under very low stresses and are expected as one of the most useful micromaterials to fabricate microsystems.

## Superplastic microforming of amorphous alloy with microdies fabricated by UV-LIGA process

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Microforming and fabrication processes are classified into two processes. The first is the transforming process of micro-shape from designing (virtual) space to the actual space and the process is correspond to 2D or 3D positioning process by using mechanical system or electron-beam controller. The second is a realization process of the shape to materials by means of etching, electroplating, milling, and etc. On the other hand, from a viewpoint of production engineering, plastic forming process is a great advantage in its productivity and enables a mass production in a controlled quality and low cost. In the forming process, the relationship between die and material is based on the difference of strength of both materials. From this point of view, superplastic materials and amorphous alloys in the supercooled liquid state have a great advantage in obtaining a deformation under very low stresses in comparison with that in the case of conventional plastic deformation, furthermore, the materials exhibit a good microformability. Then, this excellent microformability has been applied to closed die forging.

In the microforming, micro-dies and its fabrication processes are very important. In the present paper, UV-LIGA process is applied to the microfabrication of micro-dies. A negative-tone, near-UV resist SU-8 was used for photolithographic fabrication of microgear models. Number of teeth is 12 and gear module is 10 micrometers, since, the diameter of the pitch circle is 120 micrometers. Micro-dies for superplastic forging were fabricated by subsequent electroforming of Ni. With the micro-dies, micro-forging was carried out with specially developed forging machine. The apparatus facilitate a micro-furnace and its PID temperature controller, loading mechanism with electromagnetic linear actuator, and measurement and control unit with a microcomputer. The machine is subjected to a vacuum or Ar gas atmosphere in a chamber.

The selected material was Pd40Cd30Ni10P20 amorphous alloy. The material exhibits a complete Newtonian viscous flow at temperatures between 577K and 673K in the supercooled liquid region and furthermore excellent microformability in a submicrometer scale. The specimen is placed in the forging machine and worked at temperature of 640K under a compressive load of 24N (8 MPa in applied mean stress) to a microgear of 10 micrometers in module. These processes are confirmed to be useful for fabrication of microcomponents of amorphous alloys.

## Superplastic extrusion of microgear shaft with photochemically machinable glass dies

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This study seeks to establish microforming technologies for microsystems parts such as microgears of under 50 micrometers in module. For this purpose, it is important to develop new methods to fabricate micro-dies and to select suitable materials for microforming. The extrusion forming method is used to obtain long parts that have a large aspect ratio and the same, complex shape in cross-section. This forming method is classified as either backward extrusion or forward extrusion. Photochemically machinable glass has been applied to fabricate micro-dies. A micro-gear shape is printed on a photomask by electron beam lithography, and the mask is projected onto photochemically machinable glass. The glass contains photosensitive, metallic ion materials. A metallic colloid is generated by the continuous exposure of ultraviolet. This becomes a nucleus, and fine ( $\text{Li}_2\text{O-SiO}_2$ ) crystals are grown by the subsequent heating process. The solubility of these crystals in acid is up to 50 times that of the former glass, so only the exposed part is etched with weak hydrogen fluoride (HF). The exposure light can penetrate the photochemically machinable glass in the thickness direction. This enables micromachining in 2.5 dimensions and creating shapes with large aspect ratios. Superplastic forward and backward extrusion machines have been developed for the study. These machines are small enough to hold in your hand, and are useful for desktop manufacturing as microfactory cells. The specimen was placed in a container, heated, and held at the working temperature, then extruded with a piezoelectric actuator or an electromagnetic linear actuator in a superplastic or supercooled liquid state in a vacuum or an argon gas atmosphere. The working temperature of Al-78Zn alloy was 520K in superplastic condition and that of the La55Al25Ni20 amorphous Alloy was 490K in the supercooled Liquid temperature range. The module of the micro-gear shaft is from 10 to 100 micrometers and the number of teeth is from 10 to 20. In this forming process, the relation between punch stroke and punch load indicated a threshold in the punch load caused by the friction between the punch, the materials and the container. In general, the working energy is classified as deformation energy of the material and the friction energy between the material and the tool (die). In micromachining, the surface roughness and lubrication of the tool significantly affect the forming behavior.

Superplastic extrusion of microgear shaft of 10 micrometers in module proceeded under the suitable working conditions of punch load and extruding speed. The condition is determined by the characteristics of superplasticity in the materials.

## 3D wafer level packaging

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The main technical obstacle to mass production of high density modules is the challenge of forming die interconnection within a vertical stack. A manufacturer currently has to link them over the die edges. These over-edge interconnections are more art than science, and are barely automated, unreliable, and extremely cost-inefficient. A new Thru-Silicon manufacturing technology, based on Atmospheric Downstream Plasma (ADP) etching, allows for the interconnection of two or more wafers in a vertical stack. The natural etch selectivity of ADP processing allows for the simple formation of Thru-Silicon interconnections.

Thru-Silicon interconnections are formed in two major steps. The first step is to form deep metal vias on the front side of a wafer, with the vias connected to top-side circuitry. The second step is to isotropically etch bulk silicon from the backside (without a mask) to such a level that the deep Thru-Silicon vias are exposed to the backside. The ADP thinning process provides, in one step, not only thinner wafers but also back-side contacts. Process results and plans for 3D WLP stacks composed of memory, processors, control logic, and sensors for a wide variety of applications will be presented.

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## Fracture strength of GaAs wafers

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Gallium Arsenide is an attractive semiconducting material, especially in applications, where a high carrier mobility is desired. On the other hand, GaAs is extremely brittle. This brittleness is attributed either to structural defects of the as-received wafers or to microcracking

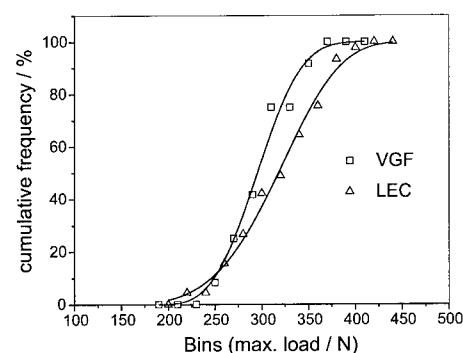


Figure 1: Cumulative Weibull plot of the fracture load of 4" LEC and VGF wafers, resp. Front surface tested.

or scratching during automated or manual wafer handling processes. Reliable wafer strength data are necessary, which represent a measure of the wafer quality as related to both structural inhomogenities caused by the crystal growth process and possible residual damage due to wafer preparation.

In the paper, the wafer strength test as realised by the authors is described and experimental results on the fracture behaviour of GaAs wafers are reported. Complete wafers were loaded to failure in a disc bending test using a ring support and a force applied to the centre of the wafer through a small diameter ball. FEM analysis were performed

in order to simulate the load-displacement behaviour and derive reliable estimates of the wafer strength from the fracture load.

The tested 4" and 6" wafers were cut from (100)-oriented single crystals grown by the liquid encapsulated Czochralski (LEC) and the vertical gradient freeze (VGF) technique, resp. Results are presented showing the large effect of details of the testing procedure as well as of the wafering processes on the fracture strength. Under optimised testing conditions a fracture load of at least 200N was measured for standard high quality (100) oriented wafers (Fig.1), which corresponds to a fracture strength exceeding 1,000MPa. This figure is significantly higher than corresponding data derived from four-point bending experiments reported in the literature [1]. No difference in the breakage behaviour between 4" LEC- and VGF wafers has been observed. A fracture mechanical interpretation of the data will be given using results of additionally performed fracture toughness tests.

[1] H. E. Belsinger, Jr. B. Wilner, U. Tasch, L. D. Topolski: A fracture criterion for Gallium Arsenide Wafers, Eng. Fracture Mechanics 48 (1994) 199-205.



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## New perspectives of microtribology – methods and applications

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Scanning probe microscopes (SPM) have been extensively used to study topography and mechanical properties such as elasticity on micro compound materials. Using an SPM, one can also investigate friction and performance of microsystems parts. We have employed conventional friction force microscopy (FFM) to study microscopic wear of magnetic tapes. We further developed a measurement strategy to investigate thin film lubricants commonly used in computer hard disk industry. Polymeric lubricant films of different thicknesses were measured. Depending on the probe radius and film thickness, we obtain different friction coefficients indicating altered friction mechanisms. Extending the FFM to ultrasonic frequencies, we can obtain friction maps revealing material contrast that is, unlike in conventional FFM, not influenced by the surface topography. Examples on magnetic tapes using this acoustic friction force microscope (AFFM) will be shown. With respect to MEMS, we propose the use of ultrasonic vibrations in order to reduce friction in absence of lubricants. Mechanically vibrating a silicon wafer sample in- and out-of-plane at ultrasonic frequencies can dramatically decrease the friction force as measured by both FFM and the more macroscopic pin-on-plate tester.

## Performance of high-speed diamond microswitch

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Due to its excellent material properties, diamond is an almost ideal material for applications in electronics and micromechanics. Especially due to its superior thermal conductivity diamond is considered for various purposes such as heat sinks or high power transistors. But also the mechanical properties of diamond like the high hardness, fracture strength and Young's modulus make it an interesting alternative to currently used MEMS materials like silicon. Due to the fact that the electrical conductivity of diamond may range from ideal insulating to almost metal-like conducting, fabrication of all-diamond MEMS devices is possible, significantly reducing problems normally found in multi-layer device structures, like thermally induced stress or thermal barriers.

In this work we investigate the performance of an all-diamond microswitch fabricated using a novel surface micromachining process [1]. The device structure is shown in figure 1. Due to its all-diamond structure, the heat dissipation in the device should be excellent, also the use of diamond-diamond tunneling contacts reduces the problem of sticking found in conventional microswitches using metal contacts.

The microswitch is electrostatically actuated by applying a voltage to the gate contact,

creating an electric field which bends the free standing cantilever thus closing the signal contact (fig. 1). The main device parameters determining the switching characteristics are the threshold voltage and the switch-on and switch-off times. Be-

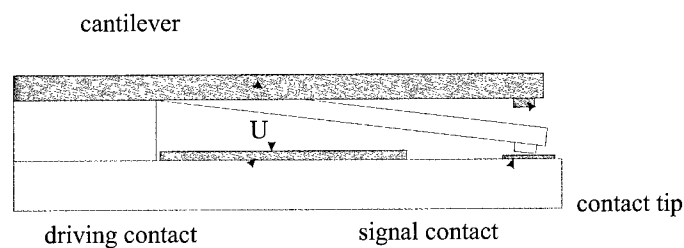


Figure 1: Cross section of all-diamond microswitch (dark: el. conducting diamond, light: insulating diamond).

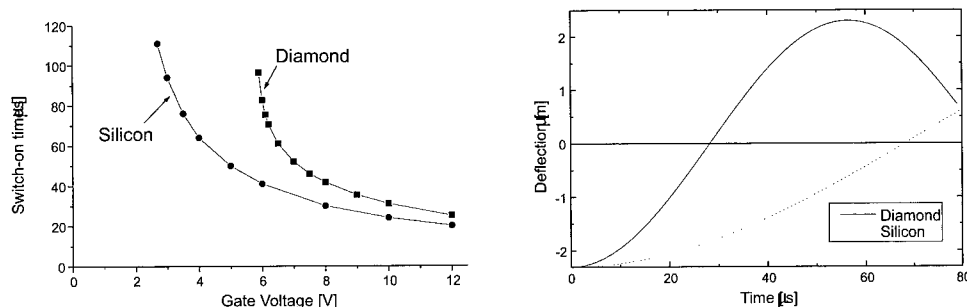


Figure 2: Calculated switch-on time (left) and switch-off characteristics (right) of diamond and silicon microswitch.

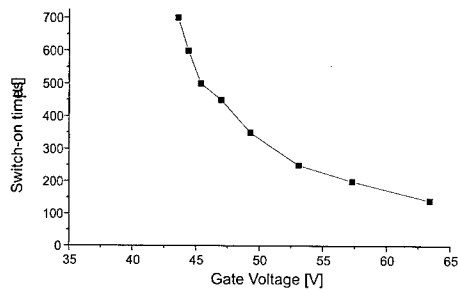


Figure 3: Measured switch-on time of diamond microswitch (different geometry).

cause of the high Young's modulus, diamond-based cantilevers exhibit a higher resonance frequency for similar device geometry compared to other materials and should thus allow operation at higher switching rates. The switching behaviour has been investigated by FEM simulations, a multi-DOF analytical model and measurements. Fig. 2 shows a calculation of the switch-on and switch-off time of a diamond and a similar silicon microswitch showing that despite of a higher threshold voltage

the high Young's modulus of diamond allows a faster recovery and thus higher switching rate for the diamond devices. Measurement results on fabricated devices will also be presented, a first result for the switch-on time of a non-optimized diamond microswitch is shown in fig. 3.

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## Nanostructured materials for microtechniques

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The reduction of the size of constructive elements requires new fabrication but also new material technologies. For this reason nanostructured materials are very interesting candidates for microoptics, microelectronics or microsystems in general. Nanostructured materials have been developed by using various technologies such as gas phase deposition methods or chemical routes. Through chemical routes, nanopowder or nanocomposite type of materials have been synthesized for the fabrication of ceramic parts by micro injection moulding, microoptical components such as optical wave guides, diffractive gratings micro lens arrays, micro fresnel lenses or laser focal lenses with very small dimensions. Processes used for the shaping are embossing, holographic techniques, photolithography or etching techniques. The state of the art demonstrates the usefulness of nanostructured materials, since the defect size can be kept at low level, and, especially for optics, the nanostructured composite materials open the opportunity for the utilization of so-called hybrid materials, where nanodispersed nanoparticles with adaptive or smart properties based on solid state physics of the nanoparticles and easily processable matrices are combined. With this technique, nanocomposite layers have been fabricated. In the paper, a review and some specific developments for the use of nanostructured materials in microtechniques are presented.

## Glob top-materials - technical demands, properties, principles of material selection

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In last time we can see many new developments in encapsulation technologies for Chip on Board-Technique. Modern packaging concepts,  $\mu$ BGA, FC, CSP needs new materials with special properties and manufacturing technologies.

It's very difficult for users to find the optimal material.

We know a lot of materials with different properties, manufacturing conditions and reliability results.

The data sheets of Glob Top-Materials and Underfillers are often however very simple and insufficient.

The processes in modern packaging technologies needs but exactly data for material selection.

The poster shows a view about the market of Glob Top-Materials, physical and technical properties.

- Glass Transition
- Coefficient of Linear Thermal Expansion
- Stress Behaviour
- Ionic Content
- Handling Conditions
- Cure Schedule
- Manufacturing Conditions

Important physical and technical properties of Glob Top-Materials were determined and compared with data sheets.

On the basis of this results follows recommends for stress minimized and reliable Glob Top-Packaging Technologies.

## Mechanical characterization of thin and ultrathin films by laser induced surface waves (Laser acoustics)

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Protecting coatings are often necessary to assure the long-term service of micro-mechanical devices. Corresponding to the small geometries the films are in the range of some hundred nanometers down to ten nanometers and below. For such thin films the reliable determination of mechanical properties is a critical problem. Laser-acoustics makes possible to evaluate the elastic behavior with high accuracy. For this evaluation the propagation behavior of laser induced ultrasonic surface waves is registered and compared with the theoretical predictions. By a fully automatic fitting procedure the sought film parameters as Young's modulus, density or thickness are derived. The number of independently determinable quantities depends on the non-linearity of the dispersion curve, i.e. the frequency dependence of wave velocity.

The correlation of Young's modulus with atomic bonding (e.g. the  $sp^3$  fraction in amorphous carbon films) and with other mechanical properties (as hardness and density) is discussed. The application of laser-acoustics for the characterization of ultrathin films (down to 5 nm) is demonstrated.

## Investigation of microshaped samples of metallic materials and carbon fibers

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To identify mechanical properties of materials used in the field of microsystemtechnology, a tensile testing machine (loading frame) was developed in the Institute of Material Research at Forschungszentrum Karlsruhe. This loading frame, installed in a standard single screw testing machine allows testing of microshaped samples as well as samples of very small masses with a diameter down to 5  $\mu\text{m}$ . The alignment between sample axis and loading axis is better than 3  $\mu\text{m}$  whereas their angularity is better than 1/10 of a degree. This system enables tensile and creep tests to be performed under constant load, strain and displacement control. In tensile tests with unloading paths the measurement of the Young's modulus is possible. The resolution of the signals is in the range of mN for load, and 0.1 mm for displacement.

In a second step a torsion drive was integrated into this loading frame, which allows tensile-torsion experiments under very small axial constant loads. The range of the torsion velocity is  $10^3$  to  $10^{-5}$  r.p.m. The angle of twist can be measured as well as the corresponding torque down to Nm.

A wide variety of test results shows the reproducibility of the experiments. The investigated materials were Copper, Gold, Vanadium, Nickel and Austenitic Steel with typical Dimensions of 100 x 100 x 940 mm, Carbon fibers with 8  $\mu\text{m}$  diameter, as well as coated carbon fibers up to 30  $\mu\text{m}$  diameter.

The results of the experiments shows that the strength of microshaped materials is 4 times higher than of macroshaped materials, and the ductility is 3 times lower, whilst the Young's modulus remains unchanged ( materials of the same quality).

Carbon fiber material is tested as monofilaments and fiberbundles, uncoated and coated under tension and torsion. The elastic and fracture mechanical description of a fiber-matrix-cylinder, characterisation of fiber, matrix and interface, influence of the interaction fiber - composite, and thermal treatment will be used for the verification of a damage model.

**Key-words:** microsystemstechnology, loading frame, torsion drive, mechanical properties, damage model,

## Preparation and modeling of fine-scaled 1-3 piezoelectric composites

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1-3 composites of piezoelectric rods embedded in a passive polymer matrix show superior properties for ultrasonic transducer applications. They combine, for example a high coupling coefficient and low acoustic impedance. Improved functional properties like higher bandwidth and working frequency can be expected by downscaling the diameter of the piezoceramic rods and varying their shape and arrangement, as well.

In view of new applications in biomedicine and non destructive testing of materials at least two tasks have to be solved: the development of a cost effective technology for the preparation of fine scaled 1-3 composites as well as the development of modeling for being able to design usefull patterns for specific application. The present paper gives a summary of new results based on piezoceramic microstructures and their perspectives in ultrasonic applications.

The here considered approach of preparation, called soft mold technology, uses silicon mastermolds, which are structured by micromachinig like dicing or ion etching (ASE™). This allows a high variety of rod sizes, shapes, spacings and arrangements. The combination of Si-microstructuring and ceramic molding is possible through soft plastic templates, which are reusable. As PZT-material a commercial soft ceramic powder has been used, giving grain sizes of 0.5 – 3,5 µm and a densitiy of 98 % within the sintered body. The following sintered structures have been prepared, so fare : rod height: 100 - 500 µm; rod diameter: 35 - 150 µm; aspect ratio: 2:1 - 6:1.

The dielectric, piezoelectric and electromechanical properties of the composite can be described by applying an analytical approach developed by Smith and Auld and Finite Element Method modeling by Steinhausen et. al. The measured materials data were found to be in good agreement with the numerical data proposed by the two models. The future work is focused on the description of dynamic properties, as for example the calculation of the impedance spectrum. This is seen as an essential basis in the study of the correlation of composite structure and ultrasonic transducer performance.



## Breakdown of elasticity in Copper and Aluminium interconnects

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The process of replacing aluminium by copper for the metallization in advanced silicon chips has been already in full progress [1]. That is for instance because copper has a significantly higher electrical and thermal conductivity as well as a lower tendency to electromigration [2]. These are mandatory properties to be applied as material of metal interconnects in the ultra large scale integration (ULSI) technology. However, the much higher diffusivity of copper in Si, GaAs and the other usual semiconductor compounds requires additional barrier layers in comparison with aluminium. More emphasis has also to be given for electrochemical migration phenomena [3]. In this paper we want to draw the attention to the anomalous elastic behaviour and to a strong effect of stress migration in thin lines of both materials deposited on silicon which show a pronounced anisotropy.

The experiments have been performed on metal line test structures deposited on oxidized (100) Si wafers, in the case of Cu capped with a 300 - 400nm thick  $\text{SiN}_x$  passivation layer. The thickness and width of line amounted to 0.1 - 0.5  $\mu\text{m}$  and 1 - 10  $\mu\text{m}$ , respectively, in the case of Cu and to 0.35  $\mu\text{m}$  in the case of Al.

The study of the elastic and plastic behavior of metal samples is usually performed by measuring the total strain  $\epsilon$  in dependence on the applied uniaxial tensile load, where  $\epsilon$  is taken as linear combination of elastic and plastic shares,  $\epsilon^{\text{el}}$  and  $\epsilon^{\text{pl}}$ , respectively. A sophisticated procedure for studying the elasto-plastic response in the case of metal lines of dimensions as mentioned above has been the measurement of the electrical line resistance [4,5]. The equations below give the line resistance change  $\Delta R/R$  in the case of longitudinal ( $\parallel$ ) and transversal ( $\perp$ ) loading of the line by bending the whole silicon substrate:

$$(\Delta R/R)_{\parallel} = (1+2\nu+\beta) \epsilon_{\parallel}^{\text{el}} + (2-\xi_{\parallel}) \epsilon_{\parallel}^{\text{pl}} \quad \text{and} \quad (\Delta R/R)_{\perp} = (-1+\beta) \epsilon_{\perp}^{\text{el}} - (2\nu-\xi_{\perp}) \epsilon_{\perp}^{\text{pl}}.$$

The strictly positive parameters  $\nu$  and  $\beta$  are the Poisson's ratio and the product of the piezoresistivity coefficient and the Young's modulus, respectively, taken as constant for a given line material.  $\xi_{\parallel}$  and  $\xi_{\perp}$  are additional parameters describing the change of volume during the plastic deformation. Furthermore, this model includes that Hooke's law is valid for the elastic part of deformation. Then, provided that  $\nu$  and  $\beta$  are known, an analysis of  $\Delta R/R$  in dependence on  $\epsilon$  allows to separate  $\epsilon^{\text{el}}$  and  $\epsilon^{\text{pl}}$ .

It was found, that the slope of curve depends strongly on the strain rate  $\dot{\epsilon}$ . Both for Cu and for Al, at the beginning of longitudinal load the elastic deformation dominates. With increasing of bending strain pronounced plastical deformations were obtained for Al-lines in contrast to Cu. Just from the beginning of perpendicular loading strong plastic contributions of strain are present for both materials, where even the elastic part disappears for Cu-lines. That is obviously the reason for strong stress migration observed during heat treatment and in course of cyclic load.

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## Package reliability studies by experimental and numerical analysis

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Growing demands on performance, cost and the advancement in IC technology have drastically influenced packaging and interconnection technology. New packaging technologies and advanced materials have to be developed to handle larger dies, higher I/O counts, lower operating voltages, high power consumption and high clock frequencies.

Even the most efficient and reliable IC design has the potential to perform poorly if its package is improperly designed. While there are many concerns in the package design, one of the primary issues is the resulting thermo-mechanical reliability of the overall package.

An electronics package is a composite and consequently complex structure that undergoes thermo-mechanical loadings while it is being manufactured and used. Due to the thermal expansion of different package parts, strains and stresses can occur and concentrate with very large values inside the package, that can lead to material, and ultimately, component and package failure. However, over ranges of designs, processes, and material parameters, different failure modes (e.g. solder fatigue, cracking, interface delamination) are observed with significant dependence on material properties and geometry. The determination of these strains and stresses is a very difficult task because of the complex geometry and materials parameters that have to be taken into consideration. To add complexity, many of the physical characteristics of the materials used will vary with time and temperature and these are both considered to be nonlinear, making prediction much more challenging.

Some useful closed-form solutions, efficient simulation schemes, and practical experimental techniques have been obtained to get a deeper understanding of the package reliability. The state-of-the-art of electronic packaging design more and more requires direct »coupling« between simulation tools (including e.g. FE modeling) and advanced physical experiments.

Furthermore, to develop comprehensive design guidelines, numerical models and experiments have to account for all the process-induced imperfections. In general, the reliability is dependent on the type of defect, the number of such defects, the size of the defects, and the location of the defects. The models are extended, however, to account for process-induced defects, and the results obtained through the simulation and testing are used to enhance the design as well as the assembly process.

The growing use of area array packages and direct flip chip attach within harsh environments requires a high effort to guarantee sufficient reliability level. Increasing die

sizes with high I/O counts as well as increasing service temperatures present new challenges for the design of reliable packages and interconnects. The paper describes analysis results of thermo-mechanical reliability especially for flip-chip packages, for chip-on-glass packages, and of chip scale packages.

## Researching of an ordered structure of porous anodic aluminum oxides

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Now aluminum and its anodic oxide find broad applications for manufacturing of the multilevel switching boards, multilayer boards, multicrystalline modules, packages for the LSI and VLSI, electrostatic fastening systems and various gauges.

It is known, that porous structure (porosity and pore's dimensions) of penetrable materials defines their service properties.

Porosity exerts dual influence on oxide properties.

On the one hand, it gives profits by reducing of internal stress level in «film-substrate» system. On the other hand, porosity can degrade electrophysical parameters of oxide.

A most complete method of porous material structure investigation is a mercury porosimetry method. Measurements in this work were made by a porosimeter (Carlo Erba Ltd., Italy, model 2000), which was controlled by a personal computer. This let a real-time data recording during analysis and quick data processing with a resulting information on a pore's dimensions and average radius distribution.

Anodizing has been made in sulfuric, oxalic and phosphoric acid based solutions.

Results of investigation of pore's dimensions and average radius distribution dependence from an electrolyte composition are presented in this paper.

Expediency of mercury porosimetry method application for the porosity investigation of thick-film anode aluminum oxides has been proved.

## Analytic of micromaterials

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The department Environmental Engineering is dealing with environmentally beneficial development of electronic products and processes. The main issues are ecological and cost aspects. Besides the reduction of energy and material the aspects of recycling will be taken into consideration, too.

The most important precondition for an environmental assessment of products are the knowledge about their material composition.

Often the industry doesn't have detailed information about the materials used in electronic components. Therefore, a chemical analysis is needed.

The analysis procedure of electronics components (including connections and base materials) will be represented in detail in this paper.

From the chemist's point of view electronic components are very complex mixtures of substances made of different kinds of tight composite materials, which have to be separated for the quantitative analysis. To save costs very often estimations are applied.

In the next step the analysis methods necessary for the complete qualitative assessment of material contents will be introduced:

The organic materials in the components are investigated using the Fourier-Transformation-Infrared (FTIR)-Spectroscopy combined with different accessory devices. Besides the classical kaliumbromide- and horizontal ATR method in particular the combination of a FTIR-Spectroscope and a microscope offers a very quick and simple method. The special »Grazing Angle« accessory facilitates examinations of very thin organic layers over metal.

LLA Umwelttechnische Analytik und Anlagen GmbH developed a laser-induced plasma analysator (LIPAN 3000M) for metal detection on printed circuit boards, especially in fine metallic structures. This method facilitates detecting almost all heavy metals without further sample preparation within few seconds. For frequent and similar analysing tasks it is even worth while conducting expensive calibrations for quantitative analyses.

## Estimation of curing degree on epoxy resin systems by FTIR

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In the microelectronic sector epoxy resins are used as encapsulation materials in the chip on board technique, for transfer mold encapsulation and as underfiller in the flip chip technique.

The mechanical properties of cured epoxies change strongly in dependence on the postcuring time. It is supposed that these changes are caused by a superior influence of the chemical post cross-linking.

In this context, users remarked that the curing-time-determination of epoxy resins by dynamic scanning calorimetry (DSC) gives no satisfying results.

In this paper, the Fourier-Transformation-Infrared-(FTIR)-Spectroscopy will be described as a successful method to determine the degree of cross-linking in epoxy resin / hardener systems.

For the measurement a combination of FTIR-spectroscope and a microscope were used to be able to analyse the materials fast and without sample preparation.

Up to now, it was difficult analysing the original material with Fourier-Transformation-Infrared-(FTIR)-Spectroscopy. The high content of filler in epoxy resins made it impossible to interpret spectra in the fingerprint area.

Therefore we developed a special preparation technique to separate the disturbing filling materials prior to measurement.

Exemplary a filled epoxy resin for the transfer mold encapsulation was analysed in state of producer delivery. After having separated the fillers  $\text{SiO}_2$  and soot, it was possible to receive a interpretable spectra.

The disappearing of the characteristic epoxy band in FTIR-spectra shows the state of cross linking of the material after curing the resin.

In co-operation with the industry we measured a filled epoxy resin and a unfilled reference resin with the same chemical composition. Thus, e.g. solvent influences on the resin / hardener system could be excluded.

## Lyotropic liquid crystalline polymers – a new thermotropic material

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In the last decade, thermotropic materials (for example polymer blends or aqueous gels) have met with growing interest [1].

The aim of this work was to examine the possibility of preparing gels with new optical properties. Such materials can be used for transmittance control of Large Area Displays as well as for so-called smart or intelligent windows. These windows can regulate lighting and heating levels in buildings. Thermotropic materials combine the advantage of using solar energy for switching effects with low production costs. External energy resources are not necessary.

The aqueous gels based on lyotropic liquid crystalline phases embedded in a thermally stable polymer network show a smooth transparency – temperature profile. This is an advantage for the smart window application, where a smooth –human eye adapted – transparency change is required. The material of the polymer network combined polyvinyl alcohol (PVA) and/or polyether alcohols of different molar masses. Inorganic salts (e.g. borate complexes or alkali halogenide) added to the polymer/water mixture have a strong influence on temperature-transmission behaviour.

The results of our studies show that the detailed knowledge of the influence of the salt concentration on the temperature of the phase separation and on the temperature range in which the liquid crystalline phases exist, permits researchers to develop systems which change their transmission from clear to cloudy mode at any desired temperature [2]. It is possible to develop intelligent windows based on the presented gel systems changing their transmission values from 95% to almost 0% in the temperature range between 25 and 50°C, thus allowing them to work as intelligent shadow givers.

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## Microforging of ZrTiCuNiBe-bulk glass in the supercooled liquid state

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Bulk metallic glasses have considerable potential as advanced engineering materials for technical application due to their favourable physical and mechanical properties and to their excellent processing capabilities. In particular they are suitable for near net shaping of machine parts by die casting or by forging under low load in the low viscous state above the glass transition temperature  $T_g$ . One prerequisite for this procedure is the thermal stability of the glass in the temperature-time range for processing, because phase transitions degrade their mechanical properties. Furthermore the glass must be free of undesirable primary crystals, which disturb the forging process and degrade the quality of the forged parts.

In the present investigation the composition of ZrTiCuNiBe is optimized to get a stable bulk glass with a minimum of primary crystals. Be seems to play the key role for the glass forming ability of the ZrTiCuNiBe-bulk glasses. The frequency and size of primary crystals increases with increasing Be content. On the other hand Be is indispensable for the bulk glass formation and a higher Be-content rises the thermal stability of the glass. Thus the optimal alloy composition must be a compromise between high thermal stability of the glass and good glass quality with a minimum of primary crystals. Crystal free  $\text{Zr}_{46.8}\text{Ti}_{8.2}\text{Cu}_{7.5}\text{Ni}_{10}\text{Be}_{27.5}$  (V4)-glass rods can be produced up to 5 mm diameter, being large enough to forge microparts.

Detailed investigations were performed to elucidate the suitability of this glass for forging in the low viscous state. For this purpose the thermal stability of the glass during forging in the low viscous state is investigated by X-ray diffraction, transmission electron microscopy, differential scanning calorimetry and microhardness measurements. The creep behavior and the influence of the thermal history on the deformation behavior of the glass is studied by thermal mechanical analysis (TMA). From these measurements the temperature-time range is determined in which the glass can be forged without altering the structure significantly and without degradation of its mechanical properties. Between 643 K and 653 K, where the glass can be deformed fast enough, it remains stable for 3 h to 6 h.

Forging experiments under low load are performed. By forging with 4 MPa at 643 K in 2 h - 5 h a deformation of 20%-50% is attained. The high forging precision, necessary for near net shaping is demonstrated. Work pieces of the glass were forged in a low load, high temperature vacuum press. Examples of forged parts of mm size are presented.

## Impact of a single grain boundary and the surrounding crystallites on the electronic properties of MOS transistors

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Grain boundaries are well known to have an important impact on the electronic, thermal, mechanical and optical properties of materials. Polysilicon transistors are state of the art. However, the impact of a single grain boundary is not very well known due to the small grain size of this material. Normally hundreds or thousands of grain boundaries are involved in one transistor.

In this work we have used n-type SILSO silicon with very large crystallites with a diameter in the millimeter range. On this material we have fabricated MOS transistors so that the grain boundary is directly located underneath the gate region, therefore source and drain are located in two different crystallites. We report on the fabrication process and the electronic properties of these transistors in comparison with MOS transistors without a grain boundary in a single grain of the same material. The impact of the grain boundary and the surrounding crystallites on the electronic properties, such as output characteristic, channel mobility and other parameters will be discussed. Additionally, the preferential grain boundary diffusion of boron in n-SILSO silicon will be highlighted and is, to the best of our knowledge, reported for the first time.

## Characterisation of micro materials and micro components by combination of the micro-bending test and UNIDAC

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For increasing the reliability and lifetime of micro components and systems their behaviour under mechanical or/and thermal has to be known.

Size and geometry of the elements to be investigated make high demands on the measuring equipment. It is possible to determine and to evaluate the local displacements and displacement fields by the combination of the optical measuring procedure *UNIDAC* (Universal Deformation Analysis by Correlation), a method of digital image processing, and suitable equipments for mechanical testing.

Digitized images of the specimen under different loadings are compared by using a correlation algorithm. The deformation behaviour of material compounds and components under loading as well as further information on physical and mechanical quantities may be derived from the obtained displacement fields.

In this paper results of investigations based on a combination of *UNIDAC* and the 3-point-bending test are shown. The bending strength and the Young's modulus of materials can be determined. It is possible to pursue in-situ crack initiation and crack propagation and to investigate the interface strength interfacial and adhesion by this method.

Examples with different material compounds and components of micro system technology will be presented.

## Micromaterials topics in the German materials research program *MaTech*

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The German Federal Ministry of Education and Research (BMBF) sponsors the program **MaTech** – New Materials for Key Technologies of the 21<sup>st</sup> Century. The key technologies are information, energy, transport, medicine and tooling technologies. New materials means innovative materials tailored for new products at first referring to those key technologies and at second attractive to the international market. All materials and materials combinations fight for their market share. The program strategy and funding concept is shortly reviewed.

Examples of joint R&D-projects in **MaTech** between science and industry related to micro materials will be presented like thermal management of high frequency electronic power units, integration of passive electronic modules by low temperature cofired ceramics (LTCC), adaptronic systems, multifunctional glasses, 3D-moulded interconnect devices (MID), polymer light emitting diode (LED), polymer organic low-e materials for multichip modules.

Also the concept of competence and demonstration centers in the field of micro materials will be shortly presented.

## A study of heel crack failures in wire bonds under mechanical cycling

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Due to extended operation conditions of power electronics (high operation temperature at large vibration levels) reliability of wire bonding has become immensely important. Two kinds of wire bond failures are of main interest: a) heel crack due to bending caused by thermal expansion or by mechanical deformation of the wire, respectively, and b) bond wire lift-off due to material fatigue (in the region where the wire is bonded to the metallization) caused by shear forces which arise from different thermal expansion coefficients at the interface. The failure mode of wire lift-off has been recently studied in [1], failure mode 2 has been published in [2]. In this paper we discuss heel crack failures in Al thick wire bonds induced by cyclic lateral bond foot displacement. Such a wire deformation is equivalent to wire flexure induced by thermal expansion, or to wire flexure if the bond surfaces are relatively moved to each other under mechanical shock loading. The bond wire flexure considered here is only passive - no current is applied through the wire, hence additional bending induced by the shear forces at the interface due to heating [3] does not occur.

### Experimental

Test structures have been prepared by ultrasonic bonding (Al wire with 250µm diameter). Wires with aspect ratios (which is defined as the ratio between loop height and loop width) ranging from 0.1 to 0.5 and widths from 5-10mm are bonded between PCB-Cu laminates and DCB-substrates. An accelerated test has been developed where the displacement of the bond foot in the direction of the loop can be adjusted up to 50mm with an accuracy of 1mm. For a given displacement, testing of wires with different aspect ratios results in different stress levels at the heels and as a consequence, different cycles to failure (lifetimes) are obtained.

Cyclic displacement has been applied with repetition frequencies of 7-13Hz at different amplitudes (displacement of 5-50mm). The occurrence of a heel crack failure is detected by an optical microscope. The damaged region is further analysed by electron microscopy.

The number of cycles to failure is found to strongly depend on the geometry of the wire: the higher the loop the higher the number of cycles to failure. A variation of the aspect ratio from 0.14 to 0.21 gives an increase in the number of cycles to failure (life time) by a factor of 10.

### Modeling

Based on the model introduced in [3] the deformation of the wire and the mechanical stresses along the wire length with the geometry data and the displacement from the

experiment are analysed by Finite Element Modeling [4], where the elastic and plastic wire properties are accounted for. Regions of maximum stresses revealed by the numerical model coincide with regions where heel cracks are initiated. Furthermore, a lifetime criterion for the bond wire based on a Coffin-Manson law is derived.

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## Characterization of erbium-doped titania films synthesised by sol-gel method on porous alumina

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Titanium dioxide films doped with erbium were fabricated by spin-on sol-gel technique on porous anodic alumina from  $\text{Ti}(\text{OC}_2\text{H}_5)_4$  precursor. The samples subjected to the thermal processing up to 1270 K exhibit strong luminescence at 1.53  $\mu\text{m}$  associated with  $^4\text{I}_{13/2} - ^4\text{I}_{15/2}$  transitions of  $\text{Er}^{3+}$  ions in  $\text{TiO}_2$  xerogel observed at room-, liquid nitrogen- and liquid helium temperatures. The intensity of photoluminescence increases with the number of xerogel films deposited onto alumina. SIMS analyses shows that the pores of anodic alumina are filled out by the xerogel after sequential spinning of ten layers. The luminescing xerogel films reveal high value of refractive index (2.4) and may attract significant interest towards the planar erbium doped waveguides.

## Properties and integration study of oxazole dielectric OxD

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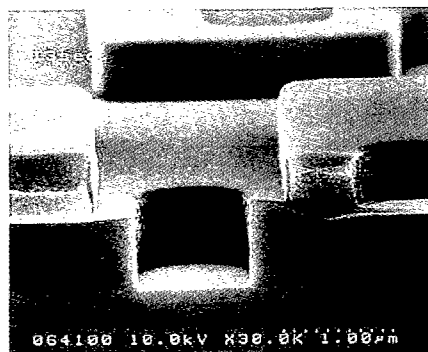
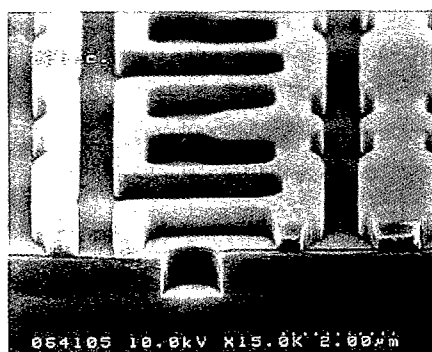
This paper presents the study of the organic low-k material Oxazole Dielectric "OxD" including its thermal, chemical, electrical and mechanical properties. The suitability of OxD for a damascene architecture has been assessed by investigating its properties after various plasma treatments and by integration of OxD with copper.

The precursor of OxD is a poly(o-hydroxy)amide which is converted to Oxazole Dielectric by thermal cure at  $T = 350\text{ }^{\circ}\text{C}$ . The dielectric layer adheres excellently to PECVD- $\text{SiO}_2$  and  $\text{-SiN}$  as well as to TaN and TiN. Thickness non-uniformities are better than 1% across a 150 mm wafer. Typical stress values at  $25\text{ }^{\circ}\text{C}$  are 35 MPa and the layers are thermally stable up to  $500\text{ }^{\circ}\text{C}$ . The dielectric constant  $k$  was determined to be 2.5 by means of impedance spectroscopy. The  $k$ -value is almost independent of frequency in the range of up to 10 MHz and it does not change after temperature cycling at  $450\text{ }^{\circ}\text{C}$ .

GC/MS investigations at  $400\text{ }^{\circ}\text{C}$  showed only low amount of outgassing molecules which do not influence the properties of OxD after being integrated into the damascene structure. The impact of Ar,  $\text{CF}_4$  and  $\text{O}_2$  plasma treatments on the bulk and surface composition and properties of OxD were determined by XPS spectra and Atomic Force Microscopy.

The patterning of OxD is feasible for Single and Dual Damascene architectures. The figure reveals a 700 nm deep via beneath a 500 nm deep trench which was patterned using the buried hardmask approach by the use of  $\text{SiO}_2$  hardmask and  $\text{O}_2$  plasma etch-

SEMs of Dual Damascene architecture patterned into OxD





ing. The SEMs demonstrate the excellent selectivity of the OxD patterning process towards  $\text{SiO}_2$ . Vias and trenches show smooth sidewalls and bottoms. The dimension control of both features is perfect.

Electrical characteristics of copper lines (linewidth 500 nm, spacing 300 nm), embedded in OxD after CMP processing, were investigated. Meander line resistances as well as leakage current measurements between unpassivated copper comb and serpentine structures gave very good results. These results are attributed to proper control of the overall process flow, to a minimum dishing and erosion during CMP processing as well as to the fact that degradation of copper by OxD does not occur during processing.

## Materials problems in high temperature electronics

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The upper temperature limit given to most silicon devices by their manufacturers is 70°C. Military specification components are qualified to 125°C but only certain types of device are available with this certification and there are usually price and lead time penalties. Whilst operation of the silicon die itself is often possible to temperatures as high as 250°C, packaging issues limit the use of many devices to much lower levels. In fact, this aspect currently presents the greatest challenge to the successful operation of electronic components at elevated temperatures.

Vulnerable areas include die attach, contacts and increased chemical reactivity at high temperatures. Many issues remain unsolved, although progress has been made with some factors under certain conditions. A very serious issue is electromigration, whereby voids and bulges form in conductors, especially when operated at high current densities.

Increasing the size of conducting paths and reducing the current density are key to dealing with this aspect. The chemical reactivity of materials used for ohmic contacts tends to increase with temperature, leading to a need for barrier materials or alternative metals for wire bonding and contact pads.

The diffusion rate of dopants is also increased at elevated temperatures, dielectric breakdown strengths are reduced and mechanical stresses can be enhanced due to mismatched thermal properties of the various materials used in the construction of a working component. Die attach problems are often a feature of thermal expansion differentials, especially where temperature cycling is a feature of the environment.

Other materials issues relate to substrates, circuit boards, solders and adhesives. In each case, the basic high temperature functionality must be considered, along with thermal properties, chemical reactivity and ease of manufacture.

When appropriately packaged, using materials and techniques that operate successfully at high temperatures, silicon devices can address applications up to 250°C. Silicon-on-insulator (SOI) technology can extend this range to 350°C. Components based upon this technology are now used routinely for oil and gas exploration applications with a rating of 225°C. For applications above this (and for power applications above 200°C) other materials are required. This is where WBS come into their own, the three main candidates being silicon carbide, gallium nitride and diamond. Wide bandgap semiconductors (WBS) have long been promoted for use in high temperature applications. The status of technological development of these three candidate materials is

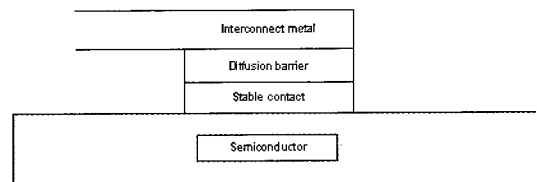


Figure 1: the barrier layer concept

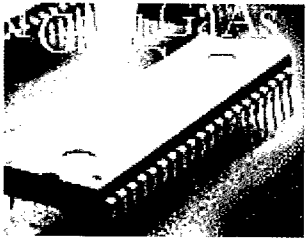


Figure 2: WBS materials

different and their varying properties offer diverse benefits, depending on the end application.

This presentation will describe the reasons why new materials offer benefits for high temperature environments, the types of device that are already seeing service and likely future developments that will increase the potential for high temperature concepts to meet requirements that current component technology cannot.

## Tensile testing of MEMS materials

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This paper presents techniques and procedures for measuring mechanical properties of MEMS materials and the latest results on the effects of size and temperature. The materials used in MEMS are actually 'new' materials in that they are on a smaller size scale than ordinary structural materials and are produced by different processes. Tensile testing is preferred, as it is for common materials, because the stress and strain states are uniform. Other approaches, such as nanoindentation tests, involve complicated stress states. Models of tiny structures where force and displacement are measured enable one to extract the properties, but the boundary conditions can be uncertain in actual practice.

Polysilicon specimens 3.5 microns thick and 600 microns wide are tested in tension by etching away the silicon substrate to release the uniform test section. The specimens are pulled in a small test machine with a linear air bearing to reduce friction, and biaxial strain is measured by laser interferometry from gold lines deposited on the specimen. Results of 48 tests of polysilicon from five production runs show a brittle material with Young's modulus =  $169 \pm 6$  GPa, Poisson's ratio =  $0.22 \pm 0.01$ , and strength =  $1.20 \pm 0.15$  GPa. Smaller specimens only 1.5 microns thick by 5 mm wide can be tested by fabricating a tensile specimen fixed to the silicon wafer on one end with a wide 'paddle' at the other end which can be gripped by electrostatic forces or glued connectors. In these cases, strain is inferred from overall displacement measurements. The scatter from these tests is naturally larger, but the material properties for polysilicon are generally the same.

Nickel microspecimens 3.1 mm long with a central test section nominally 200  $\mu$ m square are produced by electroplating into molds fabricated by the LIGA method. These specimens can be released from the substrate and placed into a small test machine. Strain is measured in this case by laser interferometry from tiny indentations in the specimen. This nickel is considerably stronger than pure nickel in bulk form; its yield strength is on the order of  $323 \pm 30$  GPa and its ultimate strength is approximately 550 GPa.

Tests are underway to measure the thermal coefficient of expansion and to explore the effects of temperature on the mechanical properties of these materials. Polysilicon is being tested at temperatures exceeding 400 °C. Silicon carbide is a promising new MEMS material because of its expected mechanical properties at high temperature and its stability in harsh atmospheres. Specimens ranging in thickness from 1 to 50  $\mu$ m will be tested in tension at temperatures up to 1000 °C, and those results will be presented.

The author acknowledges the contributions of his colleagues R. L. Edwards, K. J. Hemker, G. Coles and students K. T. Turner, K. Jackson, and A. MacAleavey. This research was supported by the National Science Foundation, the Air Force Office of Scientific Research, the Army Research Laboratory, the Naval Surface Warfare Center, and DARPA.

## Fatigue life and crack growth properties of micro-sized Ni-P amorphous alloy specimens

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Fatigue life and fatigue crack propagation tests have been performed on micro-sized Ni-P amorphous alloy specimens. The material used was a Ni-11.5wt%P amorphous thin film, which is one of the candidate materials for micromachines and MEMS (microelectromechanical system) devices. The specimens were tested in a cantilever beam type configuration of dimensions of 10 x 12 x 50 microns. The specimens were prepared by a focused ion beam machining. Notches with a depth of 3 microns were introduced in some specimens. The entire set of fatigue tests were performed using a newly developed fatigue testing machine in air at room temperature. The fatigue life curve was obtained using un-notched specimens and the fatigue strength at 500,000 cycles was determined to be approximately one-third of the static bending strength. Fatigue crack propagation tests were performed using notched specimens. Striations were observed on the fatigue fracture surfaces and fatigue crack propagation rate was estimated by the striation spacing. Size effects on fatigue strength and crack growth behaviour are discussed.

## Application of the zinc-silicat glass and $\text{GeO}_2$ thin films as diffusion sources and encapsulants for GaAs and InP

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Ion implantation is a well-established method for doping of III-V materials. However, elimination of the radiation induced damage needs some extra treatments such as encapsulation and post-implantation annealing which complicates this method.

The purpose of this work was to study Zn and Ge diffusion processes in GaAs and InP from the low-temperature zinc-silicate glass (ZSG) and  $\text{GeO}_2$  thin films, and also to evaluate their ability to serve as encapsulants during the diffusion annealing.

Thin films of ZSG and  $\text{GeO}_2$  film were produced by the low temperature ( $\sim 100^\circ\text{C}$ ) reactive ion-plasma sputtering. The single crystal Ge target was sputtered in  $\text{Ar}+\text{O}_2$  plasma for the  $\text{GeO}_2$  film deposition, while for ZSG – the composite targets consisting of Si and Zn powders were used. In case of the ZSG film deposition, regulation of Zn/Si ratio in the target yielded the desirable concentration of Zn in the ZSG film and correspondingly in the diffusion layer. ZSG/n-GaAs(InP)/ $\text{GeO}_2$  structures thus produced were subjected to the rapid thermal annealing using pulse photon source with a pulse continuity of 1-5s and different light flow powers. After these treatments Zn and Ge were diffused in the semiconductors and p-n-n<sup>+</sup> structures were obtained on GaAs and InP. Using the different Zn/Si ratios the concentration of Zn in p-layer was regulated in the range of  $10^{17} - 10^{20} \text{ cm}^{-3}$  with the layer thickness of 0.25-2.00  $\mu\text{m}$ . The fixed concentration of  $10^{19} \text{ cm}^{-3}$  was obtained for the n<sup>+</sup> layers doped with Ge.

Thus, as compared with the ion-implantation, our method simplifies Zn and Ge diffusion processes in GaAs and InP. The ZSG and  $\text{GeO}_2$  films protect GaAs and InP surfaces against As and P outflow during annealing and simultaneously serve as Zn and Ge diffusion sources producing shallow diffusion layers with the sharp p-n junction and heavily doped n<sup>+</sup> layers for ohmic contacts to semiconductors. The developed technology can be successfully used for the fabrication of devices and ICs on III-V compound semiconductors.

## Developing polymer and cement based micromaterials using reclaimed micro-balloons

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The feasibility of processing particulate composites having high stiffness to weight ratio using polymers and cenospheres and cement and cenospheres is explored in this paper. The cenospheres, being a reclaimed product from fly ash of thermal power plants, do not require any separate processing facilities, are inexpensive and use of them in a meaning full manner will reduce the environmental problems associated with their disposal. Their hollow spherical shape offers additional advantages such as, isotropy of the composite, less stress concentration, reduced overall composite density and better acoustic properties as compared to other type of reinforcements. In addition, since the spheres are inert and, usually have trapped air or CO<sub>2</sub> in side, these composites will have improved thermal insulation and fire retardant properties. The present work explores primarily the elastic and fracture properties of these composites as a function of the cenosphere content.

A simple and inexpensive technique to prepare polymer composites in the form of a functionally graded material (FGM) using polyester resin and cenospheres is developed. A nonhomogeneous distribution of the cenospheres in the polyester matrix is achieved by employing a buoyancy assisted casting process. This procedure facilitates the preparation of the composite sheets having varying cenosphere content in a single casting so that the effect of cenosphere volume fraction on the composite properties can be studied without having to prepare several sheets with different cenosphere content in each. The overall material properties of the composite are tailored by adding plasticizer to the polyester matrix. The density, quasi-static and dynamic modulus, quasi-static fracture toughness and dynamic response of the composite are obtained as a function of the cenosphere volume fraction. A fractographic analysis of the fractured specimens is also performed to identify the various fracture. The applicability of some empirical models for estimating the overall properties of the FGMs is also evaluated. Composites are also prepared using cenospheres and cement matrix.



## Giant magnetoresistance: basics and applications

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In 1988, the Giant Magnetoresistance Effect (GMR) was discovered in France in a structure comprised of two thin films of Fe separated by a thin film of Cr. Since that time many other systems have been discovered which also show the same effect, but with varying magnitudes. The fact that one could now obtain magnetoresistance values more than an order of magnitude larger than the previous maximum of 2.5% accessible in conventional bulk materials (i.e., in permalloy:  $\text{Ni}_{80}\text{Fe}_{20}$ ) represented a significant advance. The magnetic recording industry was perhaps one of the first to realize the potential of this discovery and immediately began work to implement this GMR effect into its devices. That activity has resulted in the new generation of magnetic »read heads« which are all using this effect in configurations called »spin valves«. Because of their high sensitivity and small size, these novel read heads enable information to be stored in much smaller regions on computer hard discs. As a consequence, one now sees the availability of multi-gigabit hard discs. In this presentation, a description of the effect will be reported along with an explanation of how our laboratory has prepared its world-leading spin valves. In particular, the rationale for preparing the structures in the »presence« of oxygen will be described, and future directions suggested. In addition, recent unique images of magnetic domains in a spin valve will be shown to elucidate the unusual operation of one of its key elements: the antiferromagnetic (AFM)/ferromagnetic (FM) pinning couple. Despite its discovery over 40 years ago, the origin of the AFM/FM exchange anisotropy is still controversial. The present observations clearly show that the prior methods of analysis have been incorrect. As a consequence, present and future spin valves will need to be designed with these findings in mind. As a result of this review, it will be obvious why there is so much excitement surrounding the GMR effect, and also how critical is the processing of the devices using it.

## New developments in nanomaterials

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A wide variety of scientifically and technologically interesting nanostructured materials have been synthesized and investigated during the past fifteen years. These have included metals, ceramics, and composites made by means of a number of experimental methods. While these new materials have been synthesized most elegantly from either atomic or molecular precursors, those made from bulk precursors have yielded important results as well. The structures and properties of nanostructured materials have been elucidated in a number of important areas and a fundamental understanding of the structure-property relationships is beginning to unfold. The important roles of large surface or interfacial areas and of spatial confinement on material properties, when the sizes of the nanoscale building blocks that constitute nanomaterials become smaller than the critical length scale for any particular property, have been clearly established. Investigations of the mechanical, chemical, electrical, magnetic, and optical behavior of nanostructured materials have demonstrated the possibilities to engineer the properties of these new materials through control of the sizes of their constituent building blocks and the manner in which these constituents are assembled. It is now quite clear that through such nanostructuring we can access novel material property regimes in a revolutionary manner. In this talk, an overview of the recent progress being made in our understanding of nanomaterials and their novel properties will be presented. In addition, some perspectives on future opportunities in new research directions and technological applications for nanomaterials in the 21st Century will be considered.

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## Manufacturing CSP's at waferlevel or flip chip without underfill

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The CSP (Chip Size or Chip Scale Package) is miniaturised package which is an alternative to COB (Chip on Board) and DCA (Direct Chip Attach / Flip Chip). Due to the small size of the CSP an appropriate board technology is required to allow routing. Another major issue in chip size packaging is the board level reliability, because the package has to match the different CTE's of silicon and PCB (3 ppm/k versus 16-18 ppm/k) and on contrast to flip chip technology no underfill should be required.

As the CSP is just a definition with respect to the package-to-die ratio, different types of CSP's have been developed. Main types will be presented and discussed with respect to board level reliability

For cost reasons producing CSP's at waferlevel becomes a challenging task. As no underfilling for waferlevel CSP's is required too, waferlevel CSP manufacturing practically means developing a flip chip technology without underfill.

A waferlevel CSP called S3-Diepack was developed at Technical University of Berlin. The CSP uses a solder support structure (S3) to avoid underfilling. Results from thermal cycling at board level have shown, that a reliability level can be achieved which is comparable to flex type CSP's (> 500 cycles -55 / + 125 °).

Hence a reliability level for S3-Diepack has been demonstrated which may be sufficient for consumer products.

## Application of fracture mechanics for next-generation packaging structures

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Interfacial delamination due to the presence of dissimilar material systems is one of the primary concerns in the design of electronic packages. The mismatch in the coefficients of thermal expansion between the different layers in the package can generate high interfacial stresses under thermal loads during fabrication, assembly, and/or field-use conditions. These stresses, if sufficiently large, can compromise the adhesive integrity of interfaces. The propagation of the resulting delaminations along such interfaces can degrade or completely destroy the functionality of the package. The focus of this study is to predict the potential for interfacial delamination propagation in multi-layered packaging structures. The delamination growth prediction is based on the comparison of interfacial fracture parameters obtained from numerical simulations to appropriate critical values determined experimentally using controlled fracture toughness tests.

Two-dimensional and three-dimensional numerical models were constructed of the packaging structures with interfacial cracks embedded at critical locations. The energy release rate associated with interfacial fracture was determined by employing global energy balance and a crack closure technique. The fracture mode mixity was determined using a crack surface displacement method. A material parametric study was also completed using the numerical models with pre-existing delaminations to identify material property trends that would lower the potential for failure. For a baseline materials suite, these driving-force fracture parameters were compared with experimentally determined critical interfacial fracture toughness data to ascertain the possibility of delamination growth. Based on the experiments and numerical simulations, design and processing recommendations have been developed to reduce interfacial delamination propagation in next-generation packaging structures.

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## Thin-film metal cantilever springs made by sputter-deposition stress control, with application to fine-pitch chip probing and packaging

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We have used stress control in metal film deposition to produce curved cantilever springs that protrude 10-1000  $\mu\text{m}$  above the substrate once released. The spring metal can be patterned photolithographically into arbitrary shapes prior to release, and the spring curvature and lift above the substrate can be controlled at the micron level. This brings new geometric control and new material choices to the design of three-dimensional microstructures.

Thin films deposited from the vapor phase at substrate temperatures well below their melting point grow into a quenched microstructure in which bonds can be frozen either above or below their equilibrium lengths, resulting in biaxial tensile or compressive "intrinsic" stress respectively. This is commonly observed and is generally considered a problem to be avoided by adjustment of counterbalancing process parameters. It is particularly dramatic in magnetron sputtering, where the hyperthermal kinetic energy of atoms depositing in the milliTorr range hammers the microstructure into compressive stress, whereas at higher pressures this energy is lost in gas scattering. Then the incipient crevices that develop from self-shadowing produce stretched bonds and tensile stress. The progression from compression to tension with increasing pressure is repeatable, allowing stress to be accurately controlled from zero up to over 1 GPa, corresponding to the yield point for refractory metals. A steep stress gradient normal to the substrate can thus be grown into the film, and upon undercutting of a thin underlying sacrificial release layer, the patterned film curls up to a predictable radius to relax the stress gradient in the region defined by the release etch mask. A typical spring geometry would be 1  $\mu\text{m}$  thick, 20  $\mu\text{m}$  wide, and 100  $\mu\text{m}$  long, with a 100  $\mu\text{m}$  radius, although the structure is widely scalable up and down in all dimensions.

Semiconductor chip contacting is a particularly suitable application for this technology. Shrinkage of chips is important because of operating speed, yield, cost per square cm of wafer, and packaged size. The present wire-based technologies that are used for both probing and packaging are increasingly becoming the limiting factor in chip shrinkage, because of the 60  $\mu\text{m}$  or more spacing required between bonding pads, which also are increasing in number as chip functionality increases. By comparison, we have fabricated arrays of cantilever springs on 6  $\mu\text{m}$  pitch and have flip-chip packaged Si die to them with 100% electrical yield. As with the chip wiring itself, spring pitch is limited only by the resolution of the lithography and etching. The extreme flexibility of the long, narrow springs compared to solder bumps is expected to greatly reduce joint

fatigue in thermally mismatched packages. We also have used springs patterned with sharp points to penetrate the native oxide on Al pads to make probing contactors. Here, the low scrub damage and low force per pin are significant advantages over wire needle probes.

## Micropillared layers based on porous alumina

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The peculiar inherent structure of porous anodic oxide films (AOF) of aluminium caused by the mechanism of regular, periodic microstructure formation, has attracted the attention of scientists and designers due to the practical usage of AOF in the technology of structured layers for optoelectronic devices, emissive electronics, sensors, thin-film microelectronics, etc.

For this aim, it is worthwhile to use the electrochemical anodization method as a simple and quite inexpensive one. In present paper the fabrication of pillared microstructures by means of multistep anodization of Ta/Al film system on ceramic and silicon (100) substrates is described. The results of SEM investigations of structured (pillared) layers formed by electrochemical processing of Al-Ta thin film sandwiches are presented. The basic parameters of the internal microstructure and »natural« morphology of Al anodic oxide films and pillared structured layers formed on them are given.

The structure features of porous AOF  $\text{Al}_2\text{O}_3$ , which are of interest to be obtain ordered pillared microstructures, were presented in paper. The results of SEM investigations of structured (micropillared) layers formed by electrochemical processing of Al-Ta thin film sandwiches are presented. The basic parameters of the internal microstructure and »natural« morphology of Al anodic oxide films and pillared structured layers formed on them are given. The aspect ratio for the pillars was 1,7-4,0. In the forming electrolyte the forming voltage influences on the pillar height, and the exposure time regulates the pillars diameter and the space between them.

Microstructure and composition of the combined AOF and pillar microstructures were investigated with JEM-T6 electron microscope and JSM-840 and JSM-35 scanning electron microscopes.

The essence of the transfer method of the regular structure (volume image) of Al porous oxide into another substances, consists at first in the formation of porous  $\text{Al}_2\text{O}_3$  with through pores by successive multistep electrochemical anodization of a two-layer thin-film Al-Ta composition, and then  $\text{Ta}_2\text{O}_5$  pillars are grown up by anodization of Ta film through  $\text{Al}_2\text{O}_3$  pores. At the final stage, the porous host, which served as a »natural« mask, are removed, leaving a pillared structured layer on a substrate.

To improve uniformity of distribution, stability and small scattering of characteristic element, sizes, and regularity of configuration on the large area samples the new methods of multistep electrochemical processing of Al films were developed during porous AOF formation. The possibilities of the volume image modification of microstructures are investigated also by changing of internal morphology of porous AOF. The modification (reconstruction) at the combined AOF formation is carried out by two-step anodization in two various pore-forming electrolytes.

Complex EM investigations have allowed to determine the pore depth ( $H_p$ ) and diameter ( $d$ ) in AOF, pore density ( $N$ ), porosity ( $\alpha$ ) of AOF matrixes, obtained at various

regimes and in different electrolytes, chemical and electrochemical dissolution rates, and other parameters of internal microstructure.

The aspect ratio for investigated composition was 1,7-4,0. The electrolyte composition could change the pillar sizes in almost four. The pillar radius may be varied in the 15 to 70 nm range depending on the anodization electrolyte. In the forming electrolyte the forming voltage influences the pillar height. The maximum pillar height (400 nm) and aspect ratio ( $\beta=10$ ) correspond to the maximum forming voltage 475 V. Under higher voltage the sparking (the microbreakdown of  $Ta_2O_5$ ) on the whole of substrate surface was observed. The exposure time in the forming electrolyte controlled the pillars diameter and spacing between them under usage of the same anodization electrolyte. The pillars density was determined using linear analysis method from the SEM MG's and was equal to  $(3 \cdot 10^9 \div 2,6 \cdot 10^{10}) \text{ cm}^{-2}$ . To compare, in conventional designs the elements density equals less than  $10^7 \text{ cm}^{-2}$ .

It was determined that duration of electrochemical dissolution at the pore bottom and chemical dissolution of pore walls in given electrolyte regulates the geometrical parameters of the pillar matrix structure in the tens of nanometers range. The rate of chemical dissolution, obtained from MG's, differs from the value, determined earlier by the method of zero potential (0,18 nm/min). Hence, polishing operation differs from »pure« chemical dissolution, as it is carried out at the anodization potential.



## Thermo-mechanical design support by means of FEA for advanced millimeter wave communication devices

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The design process of advanced electronic components and assemblies includes not only electrical aspects but also thermal and mechanical ones.

In the research project »Multifunctional Micrometer and Millimeter Modules (4M)«, funded by BMBF in Germany, a 28 GHz communication module was developed using a lot of plastic parts assembled with metal ones such as filter structures, distribution networks, or antenna elements. Very strong tolerances have to be held in order to save full functionality within a wide range of ambient temperature, during operation, and over the whole lifetime.

Thermomechanically optimised components have to be designed in order to fulfil these requirements. The finite element (FE) method is a suitable tool to carry out parametric studies instead of manufacturing specimens and performing time consuming experiments. Geometric details as well as the material choice can be varied. Reliability assessments and life time predictions can be derived from FE results basing on the computed stress and strain fields.

The authors explain their approach in structural optimisation of the communication module and its parts. The outstanding importance of reliable material parameters will be justified. Tensile and relaxation experiments will be pointed out which yield in temperature dependent thermomechanical material constants. Furthermore, millimeter wave circuits act well only in a dry atmosphere which yields in strong demands on the case interconnections. FE calculations were helpful to clear up the essential stress and strain relationships. An optimised interconnection design will be presented.

**Key figures:** Thermomechanical analysis, finite element methodology, material testing, structural optimisation

## Rapid prototyping of mesoscopic devices

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The trend towards miniaturization constantly diminishes the gap between conventional macroscopic manufacturing techniques (milling, turning, casting,...) and microfabrication techniques (lithography, etching, thin-film deposition, electroplating, LIGA...). Yet there still exist some distinctive differences:

- »Macrofabrication« (part or feature size usually  $>1$  mm) deals with a wide variety of materials which can be fabricated in fairly complex, three-dimensional shapes. All three dimensions are equal and there is no upper size-limit in one dimension.
- »Microfabrication« (part or feature size usually  $<100$   $\mu\text{m}$ ) allows arbitrary shapes in two dimensions (the »wafer plane«), but shapes in the third dimension usually have to be decomposed into prismatic layers. In most cases these layers cannot exceed a certain thickness (due to limited cure or irradiation depth of photoresist, limitations of the etching process or constraints in the deposition of thin films). Additionally, the number of available materials is fairly small, due to restrictions in the etching or deposition process.

In this work we show how micro- and macro-techniques can be combined in order to fabricate mesoscopic parts (part or feature size between  $100$   $\mu\text{m}$  and  $1$  mm) in a variety of materials, including ceramics, metals and polymers. The starting point for fabricating these parts are molds which are either machined conventionally or using deep plasma-etching of silicon. The applications for these parts range from engine components and electromagnetic components to parts for tooling applications.

## Semiadditive and fully additive copper deposition on ORMOCER – a comparison

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Recently different kinds of copper deposition methods on dielectric surfaces were developed and applied to test patterns regarding thin film applications of ORMOCERs (MCML/D). The aim of this work is to compare the metallization methods with respect to application-related properties of the methods and resulting test objects.

Metallization methods to be investigated are:

- 1 A semiadditive technology involving the steps sputtering Ti:W/Cu on the ORMOCER surface followed by the fabrication of a resist mask to electroplate the copper patterns to a height as necessary (common height is about 5  $\mu\text{m}$  in MCM-D), sputtering Ti as an adhesion layer, lift off Ti and stripping the resist mask, and etching of the Ti:W/Cu seed layer;
- 2 a semiadditive technology involving the pretreating which roughens and activates the ORMOCER surface by wet-chemical methods, electroless depositing full-area copper up to a height of 0.5  $\mu\text{m}$ , fabrication of a resist mask for electroplating, pulse plating up to a height of 10  $\mu\text{m}$  and etching of the Cu seed layer, and
- 3 a fully additive technology involving the steps pretreating (roughening) of the ORMOCER surface by reactive ion etching (RIE), spincoating of a precursor layer, photopatterning of the precursor layer by broad-band I-line photolithography, chemical activation of the pre-seed layer, electroless 2-step copper deposition up to a height of usually about 5  $\mu\text{m}$ .

Method (1) is universally applicable to a variety of dielectrics, which bear the thermal load during sputtering processes. It is not compatible to the common production lines of the PWB industry. The other two technologies are developed to establish the compatibility. They are more in the research state up to now.

An opportunity closer to PWB fabrication to improve the adhesion of ORMOCER layers on copper patterns is to use a wet-chemical oxidation of the copper surface to black or brown copper oxide (BCO). This technology is also applicable to the methods (2) and (3). Typical metallization results according to the three methods will be presented. Test vehicles will be compared with respect to application properties such as resolution power, shapes and regularity of test patterns, and adhesion to the ORMOCER surface. Results of test objects fabricated by IMC and TUB in the framework of the BRITE-EURAM project DONDOMMCM (continued in DONDODEM) will be presented.

## Metal/polymer interfaces and composite materials prepared by vapor phase deposition

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Metal nanoparticles dispersed within a polymeric matrix offer interesting electronic, optical and catalytic properties due to their very small dimensions [1, 2]. Such Nanocomposites are of particular interest concerning different applications, e.g. as catalytic active membranes [3, 4]. Until now the preparation of metal/polymer composites has been done mostly by adding a metal salt to a polymer solution and subsequent reduction [4] or by encapsulating of metal particles during a plasma polymerization process [5].

Metallized polymers and polymer films on metals, on the other hand, are in particular interesting for microelectronics applications. In future device generations polymers are seen as potential low-permittivity (low-k) dielectrics even for on-chip interconnects. Both processing and operation of microelectronics components involves exposure to elevated temperatures and thermal cycling. In chip applications diffusion of even very small amounts of copper, acting as a deep-level impurity, from the polymer into silicon is a concern. Therefore, much effort has been made to understand and control the interfacial microstructure and thermal stability of such interfaces. Again there are several ways to prepare the metal/polymer layers and most of them require the use of solvents.

Here we report on a completely dry process to prepare polymer films on metal substrates, metal layers on a polymer substrate and metal/polymer nanocomposite materials. Examples for each of these cases will be presented and the advantages and disadvantages compared to solution methods will be discussed for two low-k polymers, i.e., PMDA/ODA polyimide and Teflon AF and several metals, in particular copper and gold.

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## Microelectronics and fiber-optics packaging: physical design for reliability

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### **OBJECTIVES:**

- 1 Discuss, how to choose the appropriate material(s) for a particular package design and how to change, if necessary, the geometrical characteristics of the design to make a viable and reliable product.
- 2 Examine typical failure modes and mechanisms in microelectronics and fiber-optics materials and structures, and present easy-to-use formulas indicating the role of the major factors affecting their reliability.
- 3 Show how methods of Engineering Mechanics can be effectively applied to obtain closed form solutions to various practical problems of the mechanical behavior, physical design and reliability of microelectronics and fiber optics materials and structures experiencing thermally induced and/or external («mechanical») loading.

### **OUTLINE:**

- 1 *Materials, typical structures and loading conditions microelectronics and fiber-optics packaging:* materials and structures employed in microelectronics and fiber optics; current trends in microelectronics and photonics; major reliability requirements for the microelectronics and fiber optics materials and structures.
- 2 *Adhesively bonded assemblies:* thermally induced and «mechanical» stresses, bow, size effect, role of compliant attachments, adhesive strength, adhesives selection, chip-on-board assemblies, ceramic packages, «smart-card» structures, assemblies with identical adherends.
- 3 *Solder joints:* flip-chip designs in thermally matched assemblies, twist-off testing, flex circuits Vs regular substrates, selection of the underfill materials, solder materials and structures in photonics packaging.
- 4 *Thin film structures:* electronic packaging systems, semiconductor crystal growth, high-Tc superconductive films, thin films manufactured on thick (rectangular or circular) substrates, etc.
- 5 *Plastic packages of IC devices:* bow, selection of the appropriate molding compound, moisture-induced failures and their prevention, strength criteria and criteria of the molding compound propensity to moisture-induced cracking, role of fillers and delaminations, evaluation of the adhesive strength of the molding compound, «figures-of-merit» (for separation of packages that need to be «baked» and «bagged» from those that do not), etc.
- 6 *Optical fibers:* low temperature microbending; proof-testing; optical interconnects subjected to the ends off-set; fibers in termination fixtures; effect of the nonlinear behavior of silica material; optical fibers soldered into ferrules; «global» and «local»

mismatch in adhesively bonded or soldered fiber-optics structures; large deflections of optical fibers; two-point bending; fused biconical taper couplers.

*7 Dynamic response of microelectronics and fiber optics equipment:* basic principles of dynamic analysis; dynamic failure modes; response of ME and FO systems to shocks and vibrations; role of nonlinearity; shock protection of portable electronics.

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## The future of microelectronics and photonics, and the role of mechanical, materials and reliability engineering

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The emergence of, and progress in, microelectronics and photonics have revolutionized the telecommunications and information science and engineering in the 20-th century. It would be difficult to identify or even conceive of any other contemporary technologies that have had a more dramatic, pervasive, and beneficial influence on our everyday living. The extent and sophistication of the spectacular scientific and technological advances that are at our fingertips today as a result of the successes in microelectronics and photonics are amazing. These »high-technology« areas control everything - from a space shuttle to a washing machine - and are equally important from the perspective of an air force pilot, a young video enthusiast, a banking executive, an entertainer, or an homemaker. A good example is the astonishing growth of the Internet, with the number of users continuing to double every few months. Microelectronics and photonics have tremendous implications for industry, employment, strategic position of the country, and even for the future organization of the society. The well-being of the industrial nations depends on the development of intelligent products, tools and processes. It is natural that everyone, not specialists only, wonders which new applications of microelectronics and photonics are most likely to come into life in the near future and what difference these applications might make for the 21-st century home and office.

On the other hand, mechanical and materials engineering is perceived, especially by those not in the field, as the most traditional, routine, »old-fashioned« branch of engineering which simply cannot have very much in common with the advanced, sophisticated and exciting »high-technology« world. The truth is, however, that microelectronics and photonics have their foundation in materials, structures (designs) and manufacturing technologies. All the basic functions performed by electronic circuits and photonics devices are highly dependent on electrical, optical and mechanical properties of the employed materials and their reliability.

Therefore the role of a mechanical, materials and reliability engineer is extremely important in the »high-tech« world: it is his/her responsibility to make sure that the appropriate material is selected, and that the given component, device or system will be viable and reliable, i.e. that no critical failure is likely to occur during manufacturing, testing, transportation and operation of the product or the system.

In this overview we discuss the major trends in microelectronics, photonics, and some other areas of the »high-technology« engineering, with the emphasis on the role of mechanics, materials and reliability in the state-of-the-art and progress of the »high-technology«. We will describe which of the new and emerging directions and technologies look most promising, and what challenges a mechanical-materials-and-reliability engineer will most likely to encounter in connection with these technologies.

It should be pointed out that to be a »prophet« in predicting the future of the »high-technology« is not as hard as it may seem at the first glance: unless something absolutely unexpected happens, the current trends indeed determine what will take place in the foreseeable future. This is due to the tremendous momentum of these trends. Such a momentum is supported by the existing knowledge, powerful customer demand, and the enormous investments of capital and human resources.



## Review of industrial applications of CVD diamond

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Because of its remarkable range of physical and chemical properties, diamond is a unique material that can provide engineering solutions in applications where other materials have proven to be unsuitable.

The very considerable advances in CVD diamond synthesis and processing technology achieved in recent years have made available to end-users a wide range of new diamond materials which have properties tailored to specific application sectors. This has enabled novel uses for diamond in diverse and challenging fields of technology.

This paper will review the recent progress achieved in some of these applications and highlight how these have increased our understanding of some basic properties of diamond tested under extreme conditions hitherto not available. Applications to be reviewed include optical windows for high power CO<sub>2</sub> lasers, windows for megawatt power microwave beams, heat spreaders for laser diode arrays, cutting tool material that can be machined by electric discharge and radiation-hard electronic detectors for nuclear, X and γ-ray radiation.

## Thermo-graphical characterisation of the AFM cantilever

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In AFM (Atomic Force Microscopy) system the topography picture is build on the basis of signals detected from the cantilever. Then its mechanical and thermal state is strongly linked with quality and resolution of measurements.

We describe results of the experiment with application of the high resolution (10nm) Far- Field Thermography System for determination the influence of the heat generated in piezoresistive Wheatstone bridge located in the body of the AFM cantilever.

The piezoresistive sensor in AFM cantilever system consist of four piezoresistors in full Wheatstone bridge configuration. Two Wheatstone piezoresistors are placed in a longitudinal and two in a transversal direction respect to the mechanical stress when cantilever is bent by the vertical forces acting on its end. The output voltage of the Wheatstone piezoresistive bridge mainly depends on the length of the beam, cantilever width, beam thickness and average piezoresistive coefficient of the silicon. All this factors are sensitive to the local temperature of the cantilever.

Application of the Far- Field Thermographic System and, in the next step, Near- Field System are the methods for experimental study of the thermal state of the cantilever.

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## Near-field and far-field thermography in characterisation of microsystems

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We describe results of several experiments with application of the Atomic Force Microscopy (AFM) combining with high resolution Far Field Thermography system for determination the influence thermal effect of electrical field and /or electrical current on the topography of the Si microsystem structures.

In our first experiments we use AFM system to obtain the topography images of the power transistor structure. Silicon piezoresistive cantilever (length 600 mm, thickness 50 mm, and width 120 mm) is used as a nearfield detector. The topography picture was collected in contact AFM mode. The idea of this technique is based on maintaining the constant force between the tip and surface by adjusting the height of the piezoactuator.

The sample was supplied with the precise bipolar-current source with output ranges 2mA to 10mA. The topography images observed by different sample supply currents we analysed with the dedicated software *Microscan*.

In the next experiment we try to combine AFM technique with high resolution (10 mm) far field thermography system. In this manner we are able to control the behaviour of the tested microsystem in two points (input and output).

Our preliminary results confirm possibility to detect local changes in topography of the microsystems as effect of the load changes. Further experiments will be performed on temperature calibration and investigation of local thermal conductivity, microtribological properties (e.g. surface hardness and Young modulus) of the microsystems.

## Electrical conductivity model of excimer laser treaded polyimide

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Earlier investigations proved that the UV laser irradiation of polyimide is accompanied by the modification of the surface conductivity at the irradiated regions. It has been demonstrated that the electrical conductivity is increased permanently by up to 18 orders of magnitude produced by the development of a layer of carbon grains. Different properties of the excimer laser treatment can alter the electrical properties of the conductive layer. We have made experiments to study these compositional and electrical differences on the layers developed in unfilled and silver salt filled polyimide. The irradiated films were investigated by the measurements of DC conductivity in the 150-450 K temperature range, and by photoelectron spectroscopy. The conductivity mechanism of the laser treated region of polyimide is rather complex, but the main idea up to now consists of the hopping conduction, similar to the disordered, amorphous semiconductors. In this paper a more complex model of the conductive layer and the conductivity mechanism is presented.

## Fracture toughness measurements of micro-sized Ni-P amorphous alloy specimens

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Fracture toughness of micro-sized materials is considered to be different from that of bulk materials, since the surface effect on fracture mechanisms is prominent in micro-sized materials. Therefore, the fracture toughness values of micro-sized materials are extremely important to ensure the reliability of micro-sized materials when they are applied to MEMS devices. In this investigation, fracture tests have been performed for micro-sized Ni-P amorphous alloy specimens and the size effect on the fracture toughness has been examined. Cantilever beam type specimens with dimensions of 30 x 10 x 50 microns were prepared from an Ni-P amorphous thin film. Two types of notches were introduced in the specimens. One type was a mechanical notch with  $a/W=0.5$  ( $a$ : notch depth and  $W$ : width of the specimen), which was introduced by focused ion beam machining (the notch radius is 0.05 microns). The other type was a fatigue precrack which was introduced to give a final fatigue precrack length of approximately 5 microns ( $a/W=0.5$ ) using a newly developed mechanical testing machine for micro-sized specimens. Fracture tests were performed using the same mechanical testing machine. This testing machine is capable of applying static load to micro-sized specimens with a load resolution of 10 micro-N and a displacement resolution of 5 nm, respectively. Fracture of the specimens occurred in a brittle manner. The apparent fracture toughness values were calculated to be 9-15 MPam<sup>1/2</sup>, and these values were dependent on the notch type. The effects of notch radius on the fracture toughness are discussed.

## On recovery of interference fringes in holographic interferometry

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In common industrial environment, large deformation measurements of opaque bodies by means of holographic interferometry are often related to the problem of decreasing fringe spacing and contrast, causing the loss of the interference fringe pattern, which contains the whole information on the corresponding deformation. Therefore, the only way to determine the surface strain, rotation and displacement components of a structure element under load relatively to the unloaded state is first to recover the interference fringes – at least locally – and then to use the correct adequate relations to process the recovered fringe pattern properly.

This paper explicitly and quantitatively presents the general equation system for a systematic fringe recovery procedure in the general case of a large unknown object deformation. The relations for the quantitative evaluation of the recovered fringes, i.e. the optical path difference and the exact fringe vector of the modified interference pattern, are also explicitly presented. All needed relations are first introduced in form of general vector and tensor equations. Then, equations for fringe recovery are written in cartesian components and used within a quantitative experiment to demonstrate the reliability of the theory.

Some important points still should be considered in order to enable fringe recovery for general geometrical cases:

- 1 the holographic setup should allow a geometrical and/or an optical modification to adequately compensate the unknown mechanical deformation and the optical image aberrations;
- 2 the holographic method should be one of the type belonging either the real-time technique or the double exposure technique on two holograms (because not all holographic methods are suitable for this purpose);
- 3 the holographic images of the deformed and undeformed body must sufficiently overlap so that the fringe spacing must be large enough and the fringe visibility must have sufficient quality to be analysed properly.

The relations considered are general and may also be used in other application fields (with their related problems) of holographic interferometry, when the loss of fringe spacing and contrast should be compensated.

**Keywords:** Holographic interferometry, interference fringes, fringe recovery, fringe analysis, non-destructive testing, large deformation, strain measurement

## Relation between accelerated ageing tests and actual operations: Finite element simulation evaluation

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Accelerated ageing tests are widely used in electronics industry to validate the technological choices of packaging. Severe environmental and/or operating conditions make them necessary especially when significant lifetime is required. For instance, 30 years fault-free operation IGBT power modules with high blocking voltages and current ratings (e.g. 3300 V, 1200 A) are demanded for traction applications. Solder joints still remain the weakness of this packaging technology. Indeed, the Thermal Coefficient of Expansion (TCE) mismatch between the soldered materials results in cracks propagation in the solder joints which irreversibly increases the thermal resistance of the assembly and progressively leads to the failure of the module. Accelerated testing is very expensive and can last a long time, so that this kind of experiment must be optimised. Besides, thermomechanical behaviour of the assembly under realistic operating conditions for long times can be studied in shorter times thanks to finite element simulations.

This paper presents an energy-based finite element methodology aimed at designing accelerated ageing tests adapted to the environmental and operating conditions, as well as the lifetime requirement of such modules. Experiments have been carried out to validate this methodology. The same 2D axisymmetric model has been used in all simulations. It consists of a silicon chip soldered on a Direct Copper Bonding (DBC) ceramic substrate soldered on a thick copper baseplate. This typical stack is extensively used in IGBT power modules where numerous dices are connected in parallel. The model contains only one chip since the thermal coupling between the chips has been neglected. All materials except solder are supposed to deform elastically. A temperature dependent bilinear law has been used for the stress-strain curves. Moreover, a representative creep law has been used since this property plays an important role in the solder joint failure in that conditions. Connection between simulations is the energy expended in deformation which can be computed. Indeed, during the thermal cycles, the stress-strain curves follow hysteresis loops whose area represents the cyclic strain energy density. Assuming constant volume of solder, this figure provides a measurement of the cyclic fatigue damage, whatever stress-strain history. Several nodes have been selected for the energy computation, especially on the edge of the solder joint where the shear stress is the highest.

First set of simulations allows to compute the energy expended by the solder joint during the actual operation for a given time. Thermal distribution due to a representative profile has been computed in a thermal simulation. This profile is due to alternation of conducting and no-conducting phases during which power is dissipated (typically 200 W for a 50 A IGBT chip) or not respectively. Duration of each phase does not exceed 1 min typically. Then these data have been used as loading in a mechanical

simulation. The solder reflow has also been simulated to take into account the residual stresses accumulated in the solder joints. Extrapolation of these results allows to evaluate the energy for a long time ( $E_g$ ).

Energy expended during the thermal shocks is provided by a second set of mechanical simulations. For example, about fifteen thermal shocks between +125 °C and -55 °C (10 min at each temperature) have been simulated. The evolution of the energy versus the number of shocks allows to estimate how many cycles are necessary to reach the amount of energy  $E_g$  previously computed. Results have shown that about 300 thermal shocks cause damage in the solder equivalent to 30 years of operation in representative conditions. In addition, samples which have been taken as models in the previous simulations have undergone liquid-liquid thermal shocks between +125 °C and -55°C. Several solder alloy composition have been used and have shown significant cracked area after 300 cycles which has allowed to make a ranking of the different technological choices. In addition, about fifteen thermal shocks between +125°C and -15°C (10 min at each temperature and same thermal gradients than for the previous simulations) have been simulated. Temperature swing reduction of about 20 % leads to 15 % increase in the total number of cycles for the accelerated testing.

This study is a step towards the understanding of the correlation between accelerated testing and actual operating conditions. It should allow to evaluate the representativeness of these experiments in order to optimise them, and to evaluate the influence of the test parameters (dwell times, thermal gradients, temperature swing and extreme temperatures).



## Investigations and development of tactile ferropiezoelectric array sensors

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This work has presented tactile ferropiezoelectric array sensors. The nonresonance piezoelectric transformers (NRPT) with a sectional generator electrode are basic functional elements of these arrays. The running bulk acoustical wave (RBAW) has scanned the tactile effect on the array field.

Electromechanical processes in the NRPT with RBAW have been studied by the methods of mathematical modeling and simulation. Design methods for this type of tactile arrays have also been suggested. Experimental investigations of samples of these arrays have been made as well as suggestions for processing of information signals from the arrays.

The analysis of the sensors and systems for recognition of images and for tactile sensing, established on ferroelectric ceramics and polymers, has given reason for the choice of NRPT with RBAW as a primary functional element of the developed tactile array.

Two- and one-dimensional mathematical models taking into account the special features of the NRPT with RBAW describe the electromechanical processes in the functional homogeneous medium of the tactile array. The two-dimensional simulation results concerning the information signals of the tactile array have permitted to define the limitations about the constructive parameters of the functional sensitive field forming the tactile array. They have determined the conditions for the substitution of the two-dimensional with the one-dimensional model, too, for the purpose of the constructive design of the tactile array. The output signal of the tactile array is investigated in this research by one-dimensional simulation. This signal depends on the parameters of the exciting voltage pulse, the characteristics of ferroceramic materials for array's substrate and constructive parameters of array field. The simulation results have provided a possibility for determining the amplitude and time-delay of the output signal as information parameters of tactile array. The requirements about these parameters have determined, too. These investigations allow lying down the criteria for estimation of tactile array's constructions and organizations.

The experimental research of the NRPT samples as array-rows with one exciting and 8 or 16 generator electrodes have provided. To prove a reliability of the mathematical models the experimental results have compared with the results of simulation. A quantitative estimation is the percentage relative error calculated for the information parameters of output signal. The expedience to process the time-delay as an information signal parameter has substantiated for recognition of tactile effect as a two-dimensional image.

Methods of constructive design of ferropiezoelectric tactile arrays with the RBAW have been suggested taking into account the possibilities of the thickfilm microelectronic

technology. It consists of the following steps:

- selection of the ferroceramic material;
- selection of the constructive variant of the sensitive field depending on the application of the array;
- constructive calculation of the tactile array and its elements;
- selection of the organization of the exciting and sensitive generator elements of the array;
- selection of the construction of the pickup electrode for the information signal.

Interfaces for primary processing of the tactile information have been offered. They can be developed depending on the array's organization and the determined criteria about the input-output channels, the operating time for the registration of the information, the sensitivity and identification of the tactile image. The problems of multiplication of the sensitive field size and element number have been discussed with the purpose of the enlarging the functional possibilities of these sensors. The variants of integration of the tactile arrays in the array fields have been suggested.

## Wafer level flip chip packaging

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In last three years, flip chip technology has been increasingly employed in a variety of applications in microelectronics industry. Comparing to conventional wire bonding technology, flip chip provides lower profile, faster signal transfer, and higher I/O density. One of the key materials used in flip chip technology is the underfill encapsulant, which enhances the reliability of flip chip by more than one order of magnitude. Currently, the underfilling process is achieved at the package level, e.g. each individual chip has to be underfilled by the encapsulant after the solder reflow process. Then the encapsulant also has to be cured offline. The slow underfilling process becomes a bottleneck of flip chip technology at high production volume.

The new wafer level flip chip packaging process applies the underfill encapsulant at the wafer level before the wafer is diced into individual chips. During the assembly process, the chip is attached on to the substrate with the encapsulant on it and then is reflowed. During the reflow process, the solder connections and the encapsulant cure can be achieved simultaneously. In addition, the fluxing and flux cleaning processes are also eliminated since the underfill encapsulant possesses the fluxing function. Therefore, this wafer level underfill process reduces production time, increases production throughput, is suitable for high volume production, and significantly reduces the process cost.

A joint venture program, sponsored by the Advanced Technology Program (ATP), has been formed to explore the next generation flip chip packaging technology. In this paper, the technical challenges and the solutions for both the process verification and materials development in this program will be discussed.

## Qualification of different standard photo resins and new laser sources for micro-stereolithography

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In the area of rapid prototyping (RP), stereolithography (SL) has become one of the most important techniques with an annual growth of 30 %. Similar to other RP build-up technologies, the 3-D part is created directly layer by layer from the bottom up to the top according to cross-sections of the 3-D CAD model. However, the layer material is a liquid photo sensitive resin and its surface is scanned to cure usually either by an HeCd, Argon-ion or Nd:YVO<sub>4</sub> UV laser source. Due to the beam properties of these laser sources, the beam guiding system, the material deposition mechanism, and the resin, the smallest structures are currently limited to 100 x 100 x 50 µm<sup>3</sup>. Considering the miniaturisation tendency, there is a high demand to reduce this limitation by using improved materials, laser sources, and process techniques.

By curing standard SL materials with new laser sources, a general qualification of their influence on the minimal structure is possible. For this, 2-D linear test structures are created using epoxy based SU-8 10 (micro resist technology GmbH, Berlin) and SOMOS 7110 (alphaform GmbH, Feldkirchen) with a pulsed 355 nm (3w) Nd:YAG and a 351 nm excimer laser. To compare to UV sensitive resins, also a commercially available acrylate based photo resin for the visible range has been tested in combination with a self developed cw 480 nm Yb:Pr fibre laser. All considered laser sources have highest beam qualities, enabling spot diameters of a few microns.

The results show that independent of the material, the smallest curing structure depends above all on the beam parameters, and to the least degree on the material itself. With the considered high quality laser sources, minimal structures of less than 20 µm have been achieved corresponding to the adjusted beam shape. This proves, that current SL materials have already reached the quality to produce micro-parts. The minimal structure is currently limited by the SL equipment (laser source, beam guiding system, and material deposition system). As a consequence, it must be possible to produce micro-prototypes and micro-plastic parts with sizes of a few microns, if the beam guiding system (optical components), and the material deposition mechanism (mechanism to create thin layers) are improved and state-of-the-art laser technologies are used. In combination with innovative micro-injection molding processes, this technique has also the potential to produce micro mass production parts such as MEMS or MOEMS.

## Microstructure at indented thin GaN (0001) epilayers grown on 6H-SiC substrates

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The research activity in III-V nitride material systems is driven by their electrical and optical properties, although the structural perfection of the heterosystems is rather insufficient. In particular, the number of threading dislocations in the best nitride-based heterostructures is still in the range of  $10^{-8} \text{ cm}^{-2}$ . Recently, some papers discuss efforts to reduce the dislocation density by applying special epitaxial growth procedures like ELO, the Epitaxial Lateral Overgrowth. The results of these growth techniques are an inhomogeneous layer structure containing regions of very high and very low dislocation densities. To understand this procedure of dislocation filtering the exact knowledge of the dislocation dynamics is needed, i.e., the activation energies of nucleation and motion of the dislocations in the nitride epilayers, which are additionally dependent on the special dislocation type.

Therefore, the aim of this presentation will be to present first results about the mechanical properties of thin GaN-films (thickness of about 1  $\mu\text{m}$ ), with emphasis on their dislocation behavior with respect to hardness indentations.

First, we analyze the microstructure of our GaN/SiC heterosystems in detail by transmission electron microscopy. Dependent on of the molecular beam epitaxial growth conditions and the sensitive SiC substrate surface preparation, the GaN epilayers contain mainly threading dislocations in the lower  $10^{-8} \text{ cm}^{-2}$  range. Almost all dislocations run along the c-axis, perpendicular to the growing surface. The Burgers vectors of the dislocations were determined to be **a**, **c** and **a+c**, whereby most of the threading dislocations are screw and mixed type. This large threading dislocation density, which is actually not expected for the low lattice mismatched GaN/SiC system (3.4%), is explained by specific dislocation generation models.

Second, we present preliminary results of hardness indentations experiments. Micro-indentations with different directions of the indenter diagonals are achieved on the (0001) GaN surfaces to study the principles of plastic deformation zones in the epitaxial thin films and their active slip systems. With a series of indentations characterized by increasing load and starting with the lowest available value, we investigate the dislocation dynamics concerning the response of the pre-existing dislocations on the applied shear stress, which is caused by the indenter load, and the movement of the newly generated dislocations. These experimental data are discussed in connection with the results reported in the literature for other III-V semiconductors.

## Electrothermal interaction in power semiconductors

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Effective designs of mechatronic systems and compact power electronics with increasing volume power densities ask for minimized margin between the transient peak temperatures and the maximum ratings of the power semiconductors. In order to achieve the maximum thermal capabilities of a system, a more detailed analysis is required. Especially under extreme environmental conditions, like in automotive systems or for new assembly techniques, like the power flip chip technology, high performance simulation methods are required to obtain reliable and cost effective systems.

This paper demonstrates the advantages of different simulation models with increasing complexity for different power assembly techniques. The nonlinear behaviour of the electrical and thermal properties of the semiconductors and the complete mechanical construction are taken into account. A feedback loop is applied to consider the interaction between the electrical and thermal conditions. Resulting from this effect, the current density in the power semiconductor and the chip temperature will have a lateral and a vertical distribution and these distributions depend on the temperature itself. The resulting lateral temperatures on the chip are typically bell shaped, with their peak temperature in the center of the chip. Additionally, the current density and temperature distribution becomes time variant, depending on the electrical stimulation of the chip.

Different Finite Element Model approximations will be distinguished: a homogeneous and time independent volume power density with constant and with temperature dependent material coefficients, a non homogeneous and time variant volume power density in the power device resulting from a constant supply voltage and a time variant voltage source which causes a constant but not homogeneous volume power density. The investigations are made for standard MOS power transistor chips and for different types of assemblies on a heat sink: a standard TO220 package, a thickfilm hybrid package with an  $\text{Al}_2\text{O}_3$  ceramic, a multilayer LTCC ceramic assembly and a DCB ceramic package as used in high power applications and finally a power flip chip on  $\text{Al}_2\text{O}_3$  and LTCC substrate.

The paper emphasizes the differences between the packages with respect to the electrothermal interaction and the applied model. Temperature distributions on the chips and a comparison of the steady state and the transient temperature distribution of the different simulation models and assemblies will be given.

## A simple but powerful optical technique for monitoring crack propagation during fracture mechanical tests of polymers

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Thermosetting resins play an ever increasing role in microsystems, their main applications being molding compounds, adhesives and matrix resins for electronic switchboards. During their life time these materials have to sustain either static or dynamic mechanical stresses, as well as cycling compression-tension stresses caused by temperature cycles. Failure by brittle fracture is among the most frequent causes of failure of these materials. Therefore the increase of resistance to brittle fracture, i.e. toughening, is one of the major objectives for the development of new thermosetting resins. Such a material development has to be accompanied by fracture toughness measurements - whereby various specific requirements have to be observed:

- Fracture toughness tests have to give a maximum of information relevant for the performance of these materials during their lifetime with a minimum of testing effort,
- Additionally, in some cases this has to be achieved with only a few ten grams of material available.

Fracture of brittle (and toughened) thermosets can be described to a good approximation by linear elastic fracture mechanics (LEFM) laws. The European Group on Fracture Task Group has published an adoption of the A.S.T.M. E399-standard for fracture testing of metals to the fracture testing of polymers (*Testing Protocol*, J.G.Williams, Ed. Imperial College, London, 1990).

We have an experience of several years in fracture testing of a range of one- and two phase thermosetting resins. During this work several difficulties with the recommended practice of the ESIS protocol have been established. Especially the concentration of only one single value per test specimen, connected to the crack initiation from a natural precrack inserted by a razor blade, seems to be rather problematic, since there are significant difficulties in creating an »ideal precrack«. Observation of many cases led us to the hypothesis, that, even in case of such a precrack not being ideal, during the phase of crack propagation conditions are approaching to the ideal situation. In other words, it occurred to us that by monitoring crack propagation during such a test, a significant improvement in reliability and meaningfulness of the test can be achieved. Several principles have been applied before to monitor crack propagation. However, none of the methods reported in the literature provides simplicity (no sample preparation and no previous calibration needed) and automatic monitoring of force and crack length during the test.

With the availability of digital video cameras and powerful computers all necessary preconditions are fulfilled for the application of image analysis in daily test practice. By

using special optical filters it is possible to determine the position of the crack tip with good accuracy and reliability. This works both for transparent single phase thermosets and non-transparent two phase (toughened) thermosets. Immediately after the test (and within one minute) the crack-length versus time curve is calculated from the stored images, and together with the stored force-time-data, and subsequent LFM-analysis it is possible to get the  $K_{Ic}$  versus crack length curve (R-curve). Furthermore, in the case of stable crack propagation,  $G_{Ic}$  can be obtained by the area method of Gurney and Hunt. Due to its simplicity the new technique is well suited to be applied in chemical labs, where such materials are generally developed and a strong interest exists for fracture toughness characterisation techniques that can be easily carried out without special qualification and which nevertheless gives reliable and true material parameters. Thus, the development of this technique has been a contribution to overcome the often rather inefficient »classical work share« of the past between fracture mechanical engineering groups on the one side with no experience in material development and, on the other side, chemical departments which often apply inappropriate mechanical characterisation methods.

After a development and testing phase of about one year this paper gives a phenomenological summary of many cases of observed fracture behaviour.



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## Crack propagation behaviour in highly crosslinked polymers – a case study for a range of toughened and single phase thermosetting resins

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Both the developer and user of thermosetting resins have a strong interest in obtaining relevant material data on the resistance to brittle fracture (fracture toughness) since this is one of the key properties required both for structural and functional materials applied in microsystems.

The usual practice is to report only a single value ( $K_{Ic}$ ,  $G_{Ic}$ ) to describe fracture resistance of these materials which refers to the initiation event of a crack starting to propagate from a natural precrack.

A technique has been developed (described in another paper presented at this conference) which allows to monitor crack propagation very conveniently and automatically during a fracture mechanical test.

This technique has been applied to investigate the fracture behaviour of different classes of highly crosslinked one phase and two phase polymers (thermosets) and the results obtained cast some doubt as to the meaningfulness of reporting only a single  $K_{Ic}$  or  $G_{Ic}$  value for crack initiation. So for instance rubber toughened and thermoplastically toughened thermosets may have very similar values for crack initiation, but crack propagation occurs in a stick-slip manner in the first case and in a stable mode whereby even an increase of fracture resistance during propagation is observed in some cases. Such very different crack propagation (»R-curve«) behaviour is surely related also to quite a different mechanical behaviour of these materials when applied as adhesives or matrix resins for fibre reinforced plastics.

This paper gives several case studies of crack propagation behaviour of different thermosetting materials, and an attempt is made to classify the different types observed behaviour and relate them to structural parameters.

Furthermore, several critical items in the practice of fracture toughness measurements of thermosets are discussed and proposals are made how test practice can be improved.

## Effect of inhomogeneity on the instrumented indentation test

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Inhomogeneity plays an increasing role in micro materials testing. Ceramics with wellknown microstructure is an appropriate probe to study the effect of inhomogeneity on the instrumented indentation test. The poster demonstrates force/displacement curves recorded during the penetration of a vickers pyramid into a quartz-glass composite with grain sizes of 1.8  $\mu\text{m}$  and 20  $\mu\text{m}$  as well as porosities of 3% and 18 %, respectively. Using two types of hardness machines the indentation force ranges from 1 mN to 10 N with penetration depth from 10 nm to 10  $\mu\text{m}$ .

The main problem of such a study is the strong variability in the shape of indentation curves. Therefore a lot of tests has been performed using a defined order of indentation sites on the surface (indentation scattering). About 400 force/ displacement curves have been recorded. They are demonstrated by the appropriate plot square root of force against displacement. The slope of the curve is used for the calculation of the depth sensing hardness (or universal hardness). In this way a correlation between the shape of the indentation curve and the certain place of the indentation regarding the microstructure of the ceramics is tried to find. In addition, the effect of homogeneity is indicated by the statistical distribution of hardness.

Although the appearance of the strongly scattered force/displacement curves makes the analyse of instrumented hardness test more difficult the local strength of the microstructure can be demonstrated. The local risk to the reduction of the microstrength due to the damage generated by the sharp contact of a harder material (crack resistance) can be characterized.

## Monitoring of surface homogeneity of optical parameters of thin films

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In situ monitoring methods become a very important part of many thin film deposition techniques. In particular, optical parameters, thickness and its homogeneity belong to most required parameters of thin films to be monitored. In our group a spectroscopic reflection method for monitoring of surface homogeneity of film thickness based on light interference in thin films is being developed and tested. In this technique the dependence of the intensity of the reflected white light upon deposition time is measured. Depending on the immediate thickness of a grown (etched) thin film, the intensity of the reflected light changes. The experimental setup based on this method has been tested in a series of ex situ experiments. A tungsten lamp was used as a white light source (450 - 800 nm). The light beam ( $\phi$  10 - 15 mm) goes through the beamsplitter cube I; one part of the beam is detected in a slave channel of the fiber spectrophotometer (SD2000 Ocean Optics), the second one is reflected perpendicularly towards a substrate. After its reflection the beam goes back through the beamsplitter cube I and then is divided by the beamsplitter cube II. One light beam is detected (after passing through the set of different interference filters) by the multi-pixel detector of the CCD camera. In this way we can obtain the information about reflectance of the thin film - substrate system for the filter-selected values of wavelength at the individual points of the sample area. The second light beam is focused into a master channel of the spectrophotometer. By the numerical simulations of the spectral dependence of the reflectance measured by the spectrophotometer we can found the effective value of the index of refraction of the film over its whole illuminated area. Hence, using this value we can reveal the thickness distribution (homogeneity) over the whole film area from the reflexivity map (obtained by CCD camera). The instrument can be improved by scanning the fibers of the spectrophotometer (slave and master) over the image of the surface by means of step motors with a step accuracy of 10  $\mu$ m. Hence, we can also obtain the distribution of the index of refraction over the sample area and consequently to improve the map of the thickness.

The first results show that it is convenient to carry out the experimental procedure as follows: first, to determine the optical parameters of a thin film in several testing points by scanning the optical fibres of the dual spectrophotometer and second, to use the CCD camera as an area detector to get the map of thickness over the whole illuminated area of the investigated sample. In the contribution the ex situ results obtained by this method for thin films deposited (etched) by ion-beam methods will be presented.

## Nanostructured shock – wave consolidated intermetallics

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Many intermetallics exhibit an entire spectrum of superior mechanical, physical and chemical properties, which make them very attractive candidates for applications in severe environments (e.g. elevated temperatures, corrosive environment etc.). Unfortunately, almost all of them suffer from low ductility and fracture toughness at ambient temperature. A substantial grain refinement is commonly considered as being beneficial for an improvement of ductility and fracture toughness of intermetallics and could possibly be employed for intermetallics. The present work is a comprehensive overview of our efforts to produce nanostructured bulk intermetallics. Ball milled powders of two cubic intermetallics: B2 Fe - 45at%Al and  $L1_2$   $Al_3Ti(9at\%Mn)$ , were successfully consolidated into nearly porous - free bulk compacts by various combinations of shock wave (explosive) loading. The original ball milled powders had nanocrystalline grain size in the range of ~12 nm and ~3 nm (from XRD) for Fe - 45at%Al and  $L1_2$   $Al_3Ti(9at\%Mn)$ , respectively. The powders were completely disordered exhibiting much greater lattice parameter than that of the annealed ingots indicating the presence of antisite defects. The long range order (LRO) parameter of shock consolidated compacts was ~0.8 indicating almost full reordering upon consolidation. The lattice parameter of compacts was almost the same as that of the annealed ingot indicating a complete annihilation of antisite defects. Nanocrystalline structure was to a large extent retained in the compacts: ~20 nm and ~30 - 40 nm/ ~160 nm for Fe - 45at%Al and  $L1_2$   $Al_3Ti(9at\%Mn)$ , respectively. Interestingly, microhardness of the Fe - 45at%Al compacts was much higher than ball - milled powders. Correspondingly, their microstructure showed evidence of severe plastic deformation of fine fraction (~1 - 10 nm range) of powder particles and much less deformation of coarser particles. The microhardness of the 30 - 40 nm  $L1_2$   $Al_3Ti(9at\%Mn)$  compact (~630HV2) was almost identical as that of the ball - milled powders although that of the 160 nm  $L1_2$   $Al_3Ti(9at\%Mn)$  one was ~345HV2 being only slightly higher than the annealed ingot. Apparently, some softening occurred during shock consolidation associated with growth of nanocrystallites to ~160 nm from the initial nanosize of ~3 nm for the powders. Possibly, some unexpected temperature increase during shock consolidation led to the nanograin growth and related softening. An indentation fracture toughness was measured on compacts. The Fe - 45at%Al compacts did not exhibit any indentation microcracking up to 2000 g

load and their fracture toughness could not be estimated. Surprisingly, the indentation fracture toughness of the  $L1_2$   $Al_3Ti(9at\%Mn)$  compacts was only  $\sim 2 \text{ MPam}^{1/2}$  which can be compared with a bulk fracture toughness  $\sim 4 - 5 \text{ MPam}^{1/2}$  for a coarse - grained, as - cast ingot of  $Al_3Ti(9at\%Mn)$ .

## Characteristics of micro-metallic powders intended to be used in the production of electrochemical supplies current

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The paperwork presents the physico-chemical and technological properties of micro-metallic powders as obtained by chemical and physico-chemical methods as:

- precipitation from water solutions
- electrolysis of water solutions
- break-up of carbonyl metallic powder
- reduction of oxides by hydrogen

The use of these micro-powders in the fabrication of current supply sources is requiring a high purity, shape, size and specific surface of metallic particles, etc.

The physico-chemical characteristics of such metallic powders are also correlated with the electrochemical characteristics of the current supply sources.

Further to a comparative analysis of the physico-chemical and electrochemical parameters it is possible to recommend a specific type of metallic powder that can be used for a certain intended purpose.

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## Influence of specimen dimensions on creep behavior of solder material

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Creep and low cycle fatigue are the two most important effects for destroying micro solder joints in microelectronic components, hybrids and microsystems. In the future the solder volume will decrease further. The knowledge of the behavior of small solder volumes is therefore essential to determine their life time.

To determine the creep behavior of solder materials at high homologous temperatures viscoplastic equations can be used. The basis for these equations are creep curves achieved by tests at several stresses and temperatures.

This paper presents a comparison of the first results of the creep behavior under tension of small and large specimens of a tin lead alloy. The dimensions of the small specimens are 1 mm in diameter and a gauge length of 2.3 mm, the dimensions of the large specimens are 5 mm in diameter and a gauge length of 40 mm. The stresses are between 7 and 24 N/mm<sup>2</sup> and the homologous temperature at 0,65. The creep of the small specimens was measured free of contact with a laser-extensometer connected with a forced-air oven, and the large specimens with extensometers equipped with full bridge strain gauges.

The material shows a very small quasi-instantaneous strain response to the sudden load and no significant primary stage. The comparison of the creep curves of small and large specimens based on the secondary stage shows significantly higher creep of the small probes. The elongation of the small specimens for comparable temperatures and stresses is 10 to 30 times higher than for the large specimens. This value and the steady state strain rate depend on the load, the higher the load or stress the smaller the value.

The paper will discuss the results for the definition of the life time of small soldered joints and further investigations also on low fatigue loadings.

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## Database for materials in micro systems

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A working group of the DVM group Microsystem Technology has built up a database for materials of microsystems. The necessity of this special database is due to the fact that 90 % of the data needed for microsystems - e.g. thermo-mechanical simulation for micro solder joints - are not to be found in existing databases, books or tables, much less the dependence on thickness, process parameters and strain rate. This paper presents the created database.

Physical, chemical, and mechanical parameters, e.g. thermal conductivity, permeability, Young's modulus, fatigue behavior etc. are necessary for metals, adhesives, semiconductors, ceramics, composites, and multilayers. In spite of the very large data pool, it has to be handled by a simple and low cost database running on every PC.

The database was created with of Corel Paradox 8, an inexpensive database system for Windows NT with year 2000 compatibility. For structuring purposes it is necessary to collect data in several tables. Users can search the whole database under many aspects. These aspects could be e.g. the process technology, the function, or the thickness of the material in question.

Additional data and modifications of the database would be centrally collected and the updates will be delivered to all registered users. The next step will be an additional w interface for a password protected read access to the central database.

The existing database as presented in this paper was built up with the data given by the members of the working group. In the future it is planed to increase the amount of data pool with contribution of the customers.



## microDAC strain measurement for FEA support

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Reliability assessments basing on finite element analysis (FEA) demand a variety of input data for mechanical modeling. E.g., knowledge of time and temperature dependent constitutive material behavior, failure mechanisms and failure occurrence quite often is insufficient for microelectronics components and devices under investigation. Consequently, a strong need exists to accompany finite element simulations by experimental methods of strain and stress analysis. This approach allows to verify selected results from FEA with measurements on real components to ensure appropriate modeling. Furthermore, local strain measurement methods are a necessary tool to determine different kind of material properties.

Because of the small scale of objects and the required field data only a few methods are available for that purpose. One approach is image correlation on digital micrographs. It allows to determine strain fields at the surface of loaded objects. The paper reports the application of the corresponding microDAC tools developed at the IZM Berlin to electronics packaging problems.

It highlights the capability to study thermal deformation behavior inside such tiny structures as area array interconnects used in flip chip and chip scale packaging technology. Possible weak points of finite element modeling not taking into consideration real package constitution are demonstrated. So, neglecting inner defects at interfaces or detailed components structures, e.g. the glass fabrics in laminate PWB materials, simulation results may not reflect the real package deformation.

With regard to measurements of material properties the authors focus on the determination of coefficients of thermal expansion (CTE). Providing of appropriate CTE data for materials, like underfills, molding compounds, or polymers used in passivation and stress compensation layers, can be a crucial issue for design optimization with FEA. Traditional equipment, e.g. for thermo-mechanical analysis (TMA), runs out of availability, if small sized, soft and thin material specimens have to be analyzed. A microDAC based device allows access to these materials.

## Material characterisation of microrelays and microsensors

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Micromachined electroplated MEMS, among them microresonators, microgyroscopes and microrelays, have been rapidly developed in recent years. In addition to the development of electrolyte systems and deposition techniques and to the improvement of the technological process as well as the creation of new layouts, the comprehensive characterisation of electroplated microstructures and the applied materials becomes more and more important. For that reason, a hybrid procedure is projected applying

- virtual prototyping by Finite Element analysis (FEA) to optimise the design, to analyse the thermomechanical reliability and to develop design rules
- different experimental techniques to evaluate the material properties during the fabrication process and to determine essential material parameters.

Under this point of view the authors have developed suitable test and measuring strategies during the last years to characterise electroplated microstructures of a wide range. In accordance with the materials (Ni, NiCo, PdNi, Cu, Au, AuCo, AuNi,...) under investigation different measuring techniques chosen from an Analytic Pool can be applied:

X-ray diffraction analysis, electron beam X-ray micro analysis (EDX, WDX), autofocus method, micromap profiler, raster electron- (REM), transmission electron- (TEM), laser scanning- (LSM) and atomic force microscopy (AFM), cross correlation analysis, microDAC, micro and universal hardness tests, tensile tests, thermomechanical analysis (TMA), universal in-house measuring module applied for microfabrication techniques, thermal cycling between  $-70^{\circ}\text{C}$  and  $220^{\circ}\text{C}$ .

In the paper, selected results will be shortly presented. So, the deflection of coated wafers was analysed by autofocus measurements for different process steps and different layer systems. The geometry of electroplated structures was investigated by phase shift interferometry obtaining deviations in the local thickness of the structures. On the other hand, the electroplating process was optimised and evaluated concerning its stability by microhardness tests and surface roughness measurements by LSM. Changes in the alloy concentration (e.g. epNiCo) analysed by EDX cause other material properties like hardness and roughness. For that reason, phase, texture and stress of selected specimens were analysed by X-ray diffraction. Independent of the Co-concentration a separate Co-phase could not be proved found. By combining tensile and universal hardness tests, material parameters like the modulus of elasticity  $E$  and the Young's modulus  $\nu$  were determined at different element structures. In the case of epNi,  $E$  ( $E_{\text{Bulk}} = 189 \dots 206 \text{ GPa}$ ) varies between 110 and 200 GPa depending either on the

electrolyte used or on the technological electroplating parameters. Furthermore, the eigenfrequencies and vibration modes of different sensor designs and local stress concentrations were investigated by the FEA. In the result dynamically optimised microresonator and –gyroscope structures were suggested considering measured material parameters ( $E$ ,  $\nu$ ,  $\alpha$ ).

## Modular loading and measuring system for material characterisation of microcomponents

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The permanent increase in thermomechanical reliability becomes more and more important for acceptance and application of new microcomponents, devices, and systems. Reliability is strongly influenced by the combination of complex thermomechanical loading and, additionally, by geometric as well as physical factors caused by the technological processes (coating, soldering, bonding or assembling) which contributes to a complex failure and damage behaviour in material compounds and components. To ensure the quality of microsystems and to characterise material properties in microcomponents new nondestructive measuring methods and sensors have to be developed. The combination of experimental techniques and corresponding numerical simulations is a suitable way to improve the *thermomechanical compatibility* (TMC) and reliability starting with the design (layout) of micro systems, over to their processing technology and ending with their long term stability.

Within the government funded research project MikroMak a modular loading and optical 3d inspection system was created, tested and used to determine spatially resolved material parameters of material compounds and structured microcomponents. The measuring system consists of the following main components: loading equipment, illumination unit, microstructure under investigation, signal processing and analysing units and FE simulation tools. Different advanced optical field measurement techniques, the digital holography, the fringe projection method and the cross correlation technique, respectively, are modularly adapted so that they can be applied alternatively.

By means of virtual prototyping (3d CAD, rapid prototyping, FE simulation) an extremely stiff and deformationless heating device was developed and placed in a vacuum chamber to protect the optical path from unwanted influences especially important for digital holography. A micro-controller-based temperature measuring and controlling module allows temperature measurement up to 200 °C with an accuracy higher than  $\pm 0.5$  K. The sensitive optical inspection systems can be selectively adapted in order to realise high precision displacement measurements at the object surface in a range of 0.05  $\mu\text{m}$  up to some 100  $\mu\text{m}$ .

Various specimens consisting of different layers of typical micro materials have been measured under thermal loading. The corresponding finite element calculations resulted in numerical displacement fields which have been compared with the experi-

mental ones. Constitutive parameters in the FE model have been adapted in order to obtain good agreement. In this way, elastic moduli as well as CTE's could be determined. Furthermore, the beginning of plastic deformation could be detected for aluminium coated silicon strips under thermal influence.

## Pillared microstructures of Al and Ta anodic oxides - advanced system for display technology

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Submicrometer and nanosize regular pillared matrices present new capabilities for microelectronic and optoelectronic devices. In particular, investigations in the field of nanosized tip arrays for autoelectron emitters, field emission displays, vacuum luminescent displays and gas sensors are performed. Current trends lead to the development of a new inexpensive technique of submicrometer to nanometer pattern elements formation.

In this paper the active driving matrix fabricated by electrochemical processing of Al-Ta thin film compositions has been investigated. The matrix includes row and column buses integrated with driving pixels at the intersections. Each pixel consists of the MDM element with non-linear current-voltage characteristic, and pillared nanostructure formed on the top MDM electrode by anodizing of Al-Ta film composition and covered with deposited thin metal film. Pillars are 300–600 nm in height, 30–120 nm in diameter with density  $(2,5-5,0) \cdot 10^{10} \text{ cm}^{-2}$ . Effective pixel size is 180 micrometers. Each pillared pixel is surrounded by the row gate electrode, and the column is connected with the bottom MDM electrode.

The matrix is developed for active addressing of large-scale plasma and electroluminescent display panels.

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## Aluminium film internal stress effect on the conductor cross section being formed by anodization

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The electron-microscopic investigations show that a typical cross section of Al conductors formed by a selective porous anodization method takes on the trapezium form. Thus, a lateral drift of Al conductor dimensions under a mask by anodization is of less than the Al film thickness having been anodized up to the substrate. Proceeding from this we presume that the anodic oxidation process depends on the Al film internal mechanical stresses, the value of which is different for various directions.

An analytical dependence relating the anodization current with the mechanical stress value is derived. Based on this the expression to calculate the cross section dimension drift of the Al conductor is deduced. In the case of the Al conductor with the trapezium-form cross section it is established that a resistance value ration of the Al conductors formed on various substrates distinguished by their physicommechanical characteristics is equal to a reverse ratio of the internal mechanical stress values in the Al films. The structural stress contribution arising from the anodization process into the total internal stress values in the Al films is estimated. It is shown that for the Al films deposited at the temperatures 423 K the thermal stresses dominate due to the high difference in the linear expansion coefficient (LEC) values of the substrate and film materials. Under thermal stresses the resistance value ratio of the Al conductors is equal to the reverse ratio of LEC value difference of the Al film and substrates.

To test experimentally the proposed theoretical data using the selective porous anodization method the Al conductors were formed on the substrates of two types: (99.9%  $\text{Al}_2\text{O}_3$ ) and (100) single-crystal Si plate with a thermal  $\text{SiO}_2$  layer upon it. In terms of the conductor resistance measurement data the average resistance ratio is calculated. The obtained value utterly agrees with the calculated one, in terms of reference data. Thus it is believed that for the Al conductors being formed by the selective porous anodization method the lateral cross section is determined by the thermal stress value in the Al film.

## The aluminium film plastic deformation analysis during local anodic oxidation

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During porous anodization the internal mechanical stresses arized in the Al films and are due to Al compositional transformation into anodic  $\text{Al}_2\text{O}_3$ . The arizing structural stresses are summarized with the ones available in the Al film and may exceed an elastic limit. The Al film plastic deformation is the result of it. Thereby it is accompanied by a substance mass transfer in which dislocation slipping and creeping over mechanisms as well as partial stress relaxation play the main role.

It is shown that during local porous anodization the plastic deformation in aluminium is brought about by the edge effects of the lateral anodization under the mask proofing aluminium against the electrolyte contact.

For visual observation and subsequent quantitative estimation we propose a specific research technique of a plastic deformation region in the Al thin layers. To analyzed the plastic deformation region the Al films of 3 mkm thickness were used. A dense  $\text{Al}_2\text{O}_3$  stood duty as a mask for Al local porous anodization. Microphotographs of the structure shears at the Al/porous  $\text{Al}_2\text{O}_3$  lateral interface have been analyzed.

It is ascertained that the plastic deformation region in aluminium related with the edge effect of lateral anodization under the mask originates just from the Al/porous  $\text{Al}_2\text{O}_3$  lateral interface. Such region length in the Al film of 3 mkm thickness for an oxalic acid-based electrolyte is of 2.82 mkm at 0.08 mkm thickness. The long extension and high uniformity of the Al film plastic deformation region are due to a blocking role for the mask stresses being generated from the dense  $\text{Al}_2\text{O}_3$ .

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## Fracture mechanics characterisation of epoxy resins with Mini-Compact Tension (CT)-specimens

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Epoxy Resins are used for a wide range of applications. At room temperature crosslinked epoxies typically exhibit high modulus and near elastic stress-strain behaviour, but they have poor resistance to fracture. Complex material behaviour can often be observed when different mechanical and thermal properties clash, for example in a total sandwich, which can impair the total mechanical thermal reliability. Local defects can appear in these systems such as cracks in inhomogeneous materials produced by internal stress and the highly thermal and mechanical demands on gradients. The methods of fracture mechanics render the problems of the fatigue behaviour increasingly significant. They are seeking to optimise the toughness of epoxies under extreme conditions. As an optimum of reliability and operation safety is necessary for the industrial applications great effort was made to find geometry independent parameters. Usual compact tension specimens require many materials and are often unsuitable for experimental investigations. In the future, more is likely to be gained from methods miniaturising specimens in order to determine the fracture toughness of modified and newly developed materials by using fracture mechanics concepts.

In the present study, the influence of chemical structure parameters, test conditions (test temperature, crosshead speed) on crack resistance behaviour and fracture mechanics parameters is examined. The investigation of fracture behaviour of small specimens is based on the crack resistance analysis of standard specimens showing stable and unstable crack growth behaviour under quasi static loading conditions. Furthermore, the practical applications of fracture mechanics values on structural integrity assessments are strongly restricted by their limited transferability from specimens to components, but an extensive investigation to check out the technical limit of geometry, validity of specimen size criteria and of their thickness independence from fracture mechanics values under consideration of stress concentration was conducted. The results of experiments on standard specimens and miniature specimens were compared and existing qualitative coherence could be explained. Furthermore in this paper, the fracture properties of commercial epoxy resins filled with rigid glass particles were investigated.

## Tensile and fatigue testing for electronic materials with microforce testing system Tytron™ 250 (MTS)

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The reliability of high power and high performance packaging assembly is highly determined by thermal-mechanical properties. It is very difficult to receive material data relevant for mechanical design, reliability and for process optimising. For instance, the mechanical properties of these materials can influence on thickness. Results found in corresponding literature show a the significant difference of mechanical properties between bulk materials and thin films. For this phenomenon, more fundamental research is needed.

The Tytron™ 250 Microforce Testing System was used for investigating the behaviour of small specimens, particular in the field of electronic packaging materials and structures. The Minitester is an ideal solution for tensile testing of small specimens such as thin films and foils, also because the product components depend on temperature. Its static and dynamic capabilities make this system ideal for monotonic, durability and fatigue testing. For example, in the development of electronic products, this testsystem can simulate thermal cycling and produce fatigue data in a fraction of the time that a conventional thermal cycling tests.

The basic features of the test machine and some applications for electronic packaging materials will be presented. For example, the time-saving aspect compared to thermal cycling for determining the durability and fatigue characteristics of materials will be shown.

## Evaluation of glob top materials for chip on board (COB) applications

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Chip-on-Board - (COB) - Technology is an innovative solution for compact mounting of electronic components and is characterised by a high growth potential. Furthermore, this technology has to fulfil the demands on reliability, complexity, productivity and production economy. Filled polymers, namely glob top encapsulants, are used for this technology. Applications with high reliability requirements, sometimes combined with harsh environments, make tremendous demands on their mechanical, electrical and thermal properties. Generally, polymers are very versatile and can be used in many packaging applications and for which their mechanical properties can be changed within a wide range, resulting in materials properties ranging from rubber-like gels showing dramatic changes in modulus due to temperature changes to hard solids showing almost no change at all. Their mechanical properties often depend on temperature, degree of cure, additives, and thermal history. For the selection of assembly materials the knowledge of mechanical and thermal properties, respectively, is required. The most important properties are Young's modulus, relaxation behaviour, coefficient of thermal expansion and glass transition temperature. Many materials for glob top's of different vendors are currently in use, but the mechanical characteristics for most of these materials are either not available or insufficiently available.

The mechanical characterisation of glob top materials was adapted using classical standard uniaxial tensile test for determination of stress-strain (relaxation) curves, viscoelastic material behaviour, and Young's-Modulus at various temperatures. Their material properties such as the CTE (Coefficient of thermal expansion),  $T_g$  (glass transition temperature), dynamic storage modulus, and loss factor (tangents delta) have been measured using both thermal mechanical analysis (TMA) and dynamical mechanical analysis (DMA). The combination of material testing, material analysis and numerical modelling can bring important knowledge for their deployment.

Without a collection of information and knowledge it is not easy for the user to choose a glob top material, since the performance of these materials depends on many factors such as, among others, the curing conditions, CTE, glass transitions temperature, and modulus. However, based on the measurement results on various glob top materials, a method is proposed to find a ranking of their mechanical performance.

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## A study on rupture behavior of SMT solder joint with mechanical heterogeneity

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SMT solder joint reliability during thermal cycling was a major concern to the engineers in the field of microelectronic packaging. From the viewpoint of mechanics, solder joints were equivalent to low strength match mechanical heterogeneity. In this paper, the rupture behavior of the unsymmetrical mechanical heterogeneity was investigated using FEM modeling and thermal cycling test methods. The emphasis was put on the effect of mechanical heterogeneity on the stress-strain distribution and fracture behavior. The combination of experimental and computational results showed that stress triaxiality was the mechanical parameter governing the fracture of solder joints in the solder interlayer near the interface between ceramic chip and solder alloy, in which solder joints have severe mismatch of mechanical properties.

**Key words:** SMT solder joint, thermal cycle, finite element method, stress triaxiality

## Effect of bottom electrodes on microstructures and electrical properties of sol gel derived $\text{Pb}(\text{Zr}_{0.53}, \text{Ti}_{0.47})\text{O}_3$ thin film

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The effect of bottom electrode with Pt/Ti, Ir/IrO<sub>2</sub> and Pt/IrO<sub>2</sub> on the microstructures and electrical properties of PZT capacitors was investigated. The PZT thin films were fabricated using the addition of 15 mol% excess of Pb to the starting solution and spin coating onto Pt/Ti/SiO<sub>2</sub>/Si, Ir/IrO<sub>2</sub>/SiO<sub>2</sub>/Si, Pt/IrO<sub>2</sub>/SiO<sub>2</sub>/Si substrates. The crystalline phases as well as preferred orientations in the PZT films were investigated using X-ray diffraction analysis (XRD). The microstructure of the PZT films were studied by optical microscopy (OM) and scanning electron microscopy (SEM). The Au/Cr film was deposited by sputtering as the up electrode. The P-E hysteresis loop of these films were measured using a standardized ferroelectric test system which uses the principle of the Sawyer-Tower circuit. The dielectric constants and loss values of these films were measured at 1kHz using an impedance analyzer. The bottom electrode was found to influence the preferred orientation of the PZT films: bottom electrode with Pt/Ti or Pt/IrO<sub>2</sub> favored a (100) orientation, whereas bottom electrode with Ir/IrO<sub>2</sub> favored a (110) orientation. The remnant polarization and the coercive field of the PZT films with Pt/Ti bottom electrode were 14.1μC/cm<sup>2</sup> and 44.2kV/cm, while those of the PZT films with Ir/IrO<sub>2</sub> bottom electrode were 10.8μC/cm<sup>2</sup> and 48.2kV/cm. The PZT films with Pt/Ti bottom electrode show better ferroelectric properties. Difference between the dielectric and the ferroelectric properties in those PZT films was correlated to their microstructure.

**Keywords:** lead zirconate titanate (PZT); sol-gel; preferred orientation; Microstructure; X-ray diffraction, electrical properties

## The acceleration and deceleration effect of a crack on the stress fields at the tip of a crack

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When a crack propagates and strikes at an obstacles in the material, the crack either accelerates or decelerates depending on the properties of the obstacles. The stress fields at the tip of a crack would change as the velocity of a crack changes in such situations. Since the stress fields at the tip of a crack play an important role in determining the interaction between the obstacles and the crack, it is essential for us to derive the acceleration (or deceleration) effect on the stress fields. Such problems are not yet solved even for a crack propagating in an elastic material.

In this paper we have calculated the higher order stress fields associated with the acceleration (or deceleration) of a crack. As we calculate the fields, we find that the work of Freund and Rosakis [1], is not complete because the 1-st order stress fields at the tip of a crack diverges as the velocity of a crack approaches to zero. We have resolved this problem and confirmed that the dynamic stress fields at the tip of a crack approaches to the corresponding static stress fields, which is calculated seperately by solving biharmonic equation, as the velocity of a crack approaches zero. We then proceed to calculate the second order dynamic stress fields at the tip of a crack and numerically calculated the hoop stress at the tip of a crack. We then find an interesting differences of the stress fields at the crack-tip when the crack accelerates or decelerates.

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## Surface characterization of thermochemically polished CVD diamond films

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The surfaces of CVD diamond films were examined after various stages of thermochemical polishing. The average surface roughnesses of the as-grown films measured by the stylus profilometer were 25  $\mu\text{m}$  and 7  $\mu\text{m}$  on the growth and substrate sides respectively. Measurements with the atom force microscope revealed that the average surface roughnesses was scaled down to 2.2 and 2.1 nm on the growth and substrate sides respectively, after final fine polishing at moderate temperatures (750 °C). To our knowledge, the above figures are the best ever attained by any method used to polish as-grown CVD diamond films.

Scaling down the surface roughness of diamond films to a few nanometers is essential for the manufacturing of devices such as optical windows, transistors, pressure sensors, particle detectors etc., because it considerably reduces heat losses and facilitates firm contacts between the diamond film and external connections.

Raman spectra of the films polished at elevated temperatures (above 950 °C) showed non-diamond carbon lines in the spectra range 100 – 1600  $\text{cm}^{-1}$ . The low frequency spectra lines between 100 – 700  $\text{cm}^{-1}$  were seen for the first time in the Raman spectra of CVD diamond films and were attributed to clusters of carbon molecules left on the polished surfaces of the diamond films. The microcrystalline and nanocrystalline graphite lines at 1352  $\text{cm}^{-1}$  and 1580  $\text{cm}^{-1}$  showed an inverse relation to the two graphite lines in that its intensity was suppressed when the intensities of the two graphite lines were pronounced. No exciton lines were seen in the cathodoluminescence spectra of the films indicating that the electron-hole recombinations were either absent or below the detection limit. The only detectable defects in the diamond films were nitrogen and silicon with spectra lines at 575 nm and 738 nm respectively.

## Nanoparticles for sensors: Technology, basic understanding and applications

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Nanosciences, i.e. the physics and chemistry of structures with dimensions smaller than 100 nm at least in one direction, have emerged as one of the research areas with vast growth potential for the future. Nanotechnology has already found application in novel devices and many more are likely to follow. This is reflected by the enormous international effort not only in fundamental research but also in governmental funding and industrial input.

A promising field is the one of gas sensors based on oxide nanoparticles. It was proved that high gains in sensitivity are possible when the sensing materials are prepared in this way. However, the technology to be used is not trivial and there are many new phenomena to be taken into consideration for the complete understanding of the sensing mechanism. The challenge is worth to take cause the output could be highly specific and sensitive sensors not yet available.

SnO<sub>2</sub> sensors are the best-understood prototype of oxide-based gas sensors. That is the reason why they will be used in the contribution as model systems for showing how the research should be performed. The approach is based on a hierarchy of models (phenomenological and basic understanding ones) which allows the correlation between the sensor response in application specific conditions, the technology needed to get the appropriate sensor, and the elementary steps involved in the gas sensing reactions. In this way the complexity of the problem is described together with unifying concepts which will make possible the comprehension of gas-sensing phenomena in both the application and research domains.



## The measurement of thermal deformation in BGA assemblies using real-time holographic Interferometry<sup>†</sup>

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This study is directed toward the experimental investigation of thermal deformation of the surface mount assembly during a power cycling for the reliability analyses of the solder balls and of the thermal properties of the package materials. Two kinds of full grid BGAs (PBGA and CBGA) which are mounted on FR-4 boards are studied. A non-destructive testing method based on the Real-time Holographic Interferometry (RTHI) is used to measure the dynamic out-of-plane displacement field. The test is made in a complete power-on and power-off operation. The temperature in power on operation ranges from 25 °C to 60 °C at the center of the sample.

The results from RTHI show that the deformation of the PBGA assembly is different in the type, magnitude and the rate from those of the CBGA under the same power level. The dynamic displacement process is recorded. The ball-shape out-of-bending deformation takes place in initial stage of heating sample, and then, the saddle-shape warpage deformation has proceeded until thermal equilibrium. The deformation direction of the PBGA agrees with that of the PCB. However, a ball-shape bending deformation takes place in the CBGA during the heating load, and a saddle-shape warpage deformation only occurs in the edge of PCB. The deformation rate of the CBGA is far in excess of the PBGA one.

The causes of deformation are discussed through the analyses of the structure and the property parameters of the assemblies. This paper also analyzes the elements, i.e. the hardness of the materials, thermal conductivity, the sizes of the assemblies, etc., which influence the reliability of the assembly and solder points.

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## Application of new materials for integrated low-energy ignition elements in airbag systems - characterization and simulation

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The direction of current research activities by international producers of ignition elements for airbag systems is developing cheap elements which are possible to be integrated into an ignition microsystem. These integrated ignition element is the most important actuator in the whole airbag system.

In conformity with the integration of ignitor structure and electronic parts Si became the basic material. Related materials were selected for functional coatings.

The investigations involved preparation, characterization, testing, simulation and reliability of the functional coatings for ignition structures.

Poly-Si was used as reference material to compare our results with the literature. Furthermore hydrides of titanium, zirconium and hafnium were investigated. Optimally conversions of the metallic coatings (deposited by sputtering) after a heat treatment in a hydrogen atmosphere could be observed by X-Ray Diffraction and the nuclearphysical  $^{15}\text{N}$ -method (Figure 1).

After a heat treatment (110 °C, on air) the evaluation of stability over the time was performed by analysing the sheet resistance for TiHx- and HfHx - probes. The divergence of the resistance values related to the start value (without heat treatment) amounts 5% for TiHx and 1% for HfHx. Accordingly to [1] the thermal stability of the metal hydrides increases from Ti to Hf.

To obtain suitable design parameters for the ignition elements, a coupled electrical and

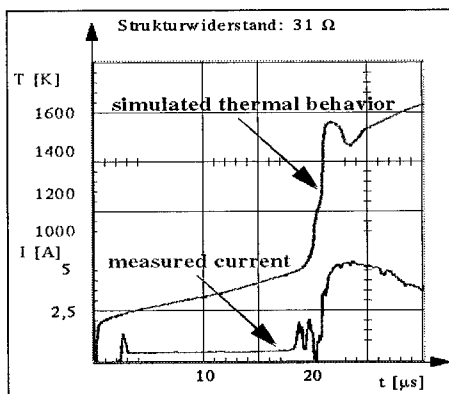


Figure 1: Hydrogendistribution in HfHx-coatings after heat treatment in a hydrogen atmosphere at various temperatures

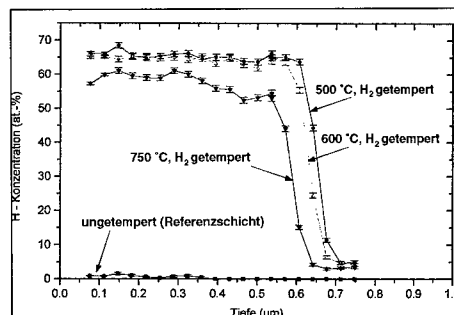


Figure 2: Simulated thermal behavior and measured current for a poly-Si structure

thermal simulation was performed by the commercial ANSYS FEM program. Figure 2 shows an example of the good agreement between the curves of the simulated thermal behavior and the measured current.

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## Patterning of SOI-materials for fabrication of resonator systems using waferbonding approaches

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Waferbonding technologies (silicon direct bonding) were used for the fabrication and packaging of micromechanical devices in bulk micromachining. Detailed investigations should show to which extent the bonding processes were able to fulfil general requirements like patterning possibilities of bonded SOI-substrates with low mechanical stress. Special high and low temperature bond processes and their integration into the technological process flow were introduced exemplarily for a gyroscope resonator system (figure 1).

The results of pattern transfer in SOI-wafers during the fabrication of gyroscope resonator structures after high and low wafer bonding processes with different pre-treatments are presented. These results are compared with measurements of surface energy of the compounds.

Our previous investigations concerning the etching behaviour of SOI-wafers have shown, that a porous intermediate oxide layer created during the bonding and annealing process leads to defects during the patterning of thermal oxide layers at the interface (figure 2), caused by an essential higher etch rate of porous layers as for the thermal oxide layers. The porous layers lead to underetched and destroyed silicon feature edges during the anisotropic etching. Further investigations showed, that bonding of an oxidised silicon wafer to a non-oxidised silicon wafer for the fabrication for SOI-wafer material reduces the influence of the porous intermediate layer (in contrast to compounds of

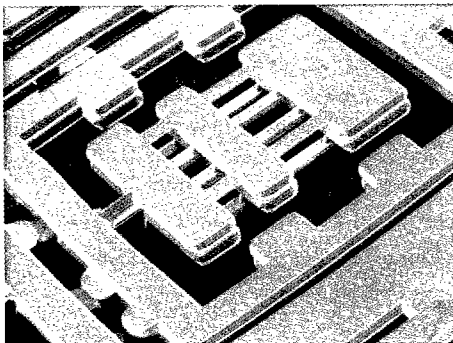


Figure 1: Silicon resonator system



Figure 2: Defects through patterning of SOI-material

two oxidised wafers). In addition it was demonstrated, that annealing at 1100°C is necessary. In this case a sufficient transfer the feature edges from one wafer to the other is achieved [1].

Current investigations are dealing with the influence of different pre-treatments and annealing temperatures of the bonded SOI-wafers on pattern transfer results and the firmness of bonded substrates.

It is found that a low temperature silicon wafer bonding approach ( $O_2$ -plasma (RIE) followed by DI-water rinsing or RCA and annealing at 400°C) delivers surface energies up to 2.8 J/m<sup>2</sup> and improves the pattern transfer quality of the SOI-wafers, and can therefore replace high temperature processes.

The application of this low temperature bonding approach allows the fabrication of silicon resonator systems of gyroscopes with a low defect level in silicon bulk micromechanics and results in a correct transfer of feature edges. Various other pre-treatments in combination with low temperature annealing achieve still supply surface energies of about 1.7 J/m<sup>2</sup>, but they lead to faulty patterning of materials.

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## Investigation of bonding behaviour of different borosilicate glasses

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These new investigations are carried out in order to characterise the anodic bonding behaviour of borosilicate glass Borofloat 33 in comparison with different suppliers of borosilicate glass wafers. The following types of glasses were involved: Borofloat 33 from Schott Jenaer Glaswerke GmbH, Corning FH-IZM from Bullen Ultrasonics Inc. (Remark: Corning FH-IZM means the glass which was supplied by Bullen Ultrasonics Inc. using the designation »Corning«), Code 7740 from Corning and SD2 from Hoya Europe B.V.

Firstly the original surfaces were characterised. This was followed by anodic bonding experiments of the glass wafers with unpatterned and patterned silicon wafers. The compounds with unpatterned substrates were evaluated and compared with respect to the parameters bonding temperature, bonding time and current flow. Furthermore, test patterns (Chevron notches) for evaluation of bond strength were prepared, anodically bonded to the glass wafers and then loaded using the Micro Chevron Test up to the failure of the bond.

All unpatterned silicon-glass compounds with the different glass types are suitable for the anodic bonding process. In case of Hoya glass type the bonding currents are significantly lower in comparison to the other glass types.

The differences in the measured fracture toughness values of the patterned silicon-glass compounds, with the exception of Hoya, is small. The tests have shown that an applied mechanical load caused cracking in the glass wafer but not in the bond interface. The tested glass type Borofloat 33 distinguishes itself by good bond behaviour with comparable bonding currents, bonding times and yield of bonded area to the other tested Glass types.

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## A constitutive material model of eutectic SnPb flip chip solder for ANSYS®

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Solder fatigue is the major reliability concern in flip chip packaging. Despite recent efforts, there is still a lack of material data for micro solder joints resulting in inaccurate life time estimations. Hence, isothermal shear tests have been carried out to determine material data of real flip chip solder joints.

A test apparatus has been designed to perform reversible shear tests on flip chip joints. In contrast to similar setups, this tester is actively compensated for its finite stiffness and features very high precision in force (1 mN resolution) and displacement (20 nm resolution measurement).

The experimental program included cyclic shear tests for elastic plastic material data as well as a new reversal creep and relaxation test procedure for time dependent material properties. FEM simulation has subsequently been applied to evaluate the experimental raw data and to extract material parameters according to the material models provided by ANSYS®.

The results of this investigation differ from published SnPb solder data. Therefore, a comparison between the published and the investigated solder data was carried out under consideration of corresponding test conditions. A correlation between data and test conditions could be found.

The paper presents the test apparatus and the adjusted ANSYS® SnPb37 models. It discusses the pros and cons of different models, the differences in material data of various sources and the effects of different models onto ANSYS® FEM results.



## Crack propagation in flip chip solder joints

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Flip chip packages may fail due to fracture of the solder joints. Although material data describing the crack propagation of solders exists, it is unknown if such data is transferable to the problem of cracks in flip chip joints. In bulk specimens the plastic zone around the crack tip can spread out freely and covers only a small fraction of the total volume. Flip chip joints are so small, that the plastic deformation zone is limited by the joint dimensions. Therefore the crack propagation behavior inside flip chip joints is supposed to be different to that of bulk specimens.

The paper presents crack propagation experiments on real flip chip specimens applied to reversible shear loading. Two specially designed micro testers will be introduced. The first tester provides very precise measurements of the force displacement hystereses. The achieved resolutions have been 1 mN for force and 20 nm for displacement. The second micro tester works similar to the first one, but is designed for in-situ experiments inside the SEM. Since it needs to be very small in size it reaches only resolutions of 10 mN and 100nm, which is sufficient to achieve equivalence to the first tester.

A cyclic triangular strain wave is used as load profile for the crack propagation experiment. The experiment was done with both machines applying equivalent specimens and load. The force displacement curve was recorded using the first micro mechanical tester. From those hysteresis, the force amplitude has been determined for every cycle. All force amplitudes are plotted versus the number of cycles in order to quantify the crack length. With the second tester, images were taken at every 10th ... 100th cycle in order to locate the crack propagation. Finally both results have been linked together for a combined quantitative and spatial description of the crack propagation in flip chip solder joints.

## Modelling of material properties for advanced packaging

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In order to predict performance and reliability of electronic components and microsystems in the development process simulations are winning ever greater importance. The accuracy of simulations depends substantially on the available materials data. Realistic result can only be achieved in thermal and thermomechanical calculations when the temperature dependencies as well as the nonlinear and time-dependent behaviour of the materials laws are taken into account.

The work reported here is dedicated to the creation of a special database for the simulation of electronic packages and assemblies. As part of this work the materials data were measured under well-defined conditions concerning test environments and state of materials (pre-conditioning). The measured original data were adapted to suitable material models. In the presentation, the results of measurements on representative and novel solder alloys, molding compounds and conductive adhesives, as well as the materials modelling for finite element simulations are presented.

As part of the work, a test procedure for the fast characterisation of solder materials was developed. Deformation and creep curves were taken up using a modified microhardness tester at different temperatures. The material properties are adapted to existing material laws by simulation.

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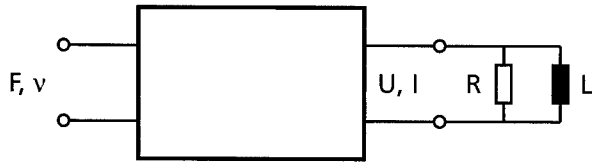
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## Electromechanical transducer and stochastic damages

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The paper focuses on the theoretical simulation of converting mechanical measurements ( $F, v$ ) into electric signals ( $U, I$ ) taking into account local microdamaging and load redistribution. A monomorph piezo bending damper



is studied to test the effect of local damaging upon the mechatronic transducer.

$$\begin{pmatrix} F \\ v \end{pmatrix} = \frac{1}{\tau} \begin{pmatrix} 1 & Z_{\text{mech}} \\ 0 & 1 \end{pmatrix} \begin{pmatrix} \tau^2 & 0 \\ 0 & 1 \end{pmatrix} \begin{pmatrix} 1 & 0 \\ i\omega C & 1 \end{pmatrix} \begin{pmatrix} U \\ I \end{pmatrix}$$

$F$  applied force  
 $v$  deflection rate  
 $Z_{\text{mech}}$  mechanical impedance  
 $\tau$  coefficient of transformation  
 $C$  capacity of the piezoelement

The cumulative, pointwise failure distribution [1] developed recently by the author,

$$D(x, y, z, Q) = \int_0^Q V_0 [1 - P(q, V_0)] \lambda(x, y, z, q) dq \quad 0 \leq D \leq 1$$

is to be characterized the local damage. The symbol  $\lambda$  defines the local failure rate in an infinitesimal subdomain at point  $(x, y, z)$ , the term in the square brackets is the survival probability of the whole volume  $V_0$  up to a current load  $q$ .

Note that the survival probability itself varies with the damage level  $D$ , likewise the mechanical impedance and the coefficient of transformation of the damper. The lecture analyses the change in damping behaviour under local failure.

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## Superhard and ultrasmooth carbon films for micromechanical components

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Many micromechanical devices contain sliding parts. For the step away from micromechanical demonstrators to real products with sufficient life-time they must be protected in a reliable manner. Such films should be hard and elastic to avoid wear, they should show low friction and high corrosion resistance and they have to be sufficient thin and smooth to preserve the original topography.

Specially prepared carbon films combine all these properties: These films achieve hardness values up to  $> 70$  GPa with elastic recovery  $> 80$  %. Even without lubricants carbon films have friction coefficients  $\leq 0,1$ . Deposited films as thin as 5 nm have demonstrated their high protection in corrosive media.

The preparation of such films requires high energies of the impinging carbon particles for the formation of strong diamond-like bonds and a low deposition temperature to avoid the relaxation of this non-equilibrium structure. These demands are fulfilled by the vacuum arc deposition in a very efficient manner. Problems arise from the difficult moving behavior of the cathode spot on carbon cathodes leading to splintering of micrometer sized particulates which may be incorporated in the growing film. These difficulties have been overcome by specially developed pulsed arc techniques (Laser-Arc, High Current Arc). For complete elimination of the remaining few particulates they are combined with magnetic filters leading to films with roughness in the nanometer scale. By modification of the deposition conditions, film properties as resistance or wettability are adapted to special application demands.

The application of these films for micromechanical devices and their protective potential for manufacturing tools in micromechanics is discussed.

## KOH etch rate and optical properties of ICPECVD silicon nitride

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Silicon nitride is an important material in MEMS technology as isolating membrane, KOH etch mask, and passivating layer. High quality silicon nitride layers can be deposited by LPCVD (low pressure chemical vapor deposition) or PECVD (plasma enhanced chemical vapor deposition) at substrate temperatures of about 750 °C or 350 °C, respectively.

Especially for sensors, however, a high quality low temperature silicon nitride is frequently needed. Therefore, some compromises have to be made with respect to quality or deposition rate and hence productivity.

We report on a new plasma deposition equipment and process which allows higher quality layers at appreciably lower temperatures. Further, we found that the layer quality can be reliably assessed by comparing the refractive index at 632,8 nm with the absorption index at 320 nm.

The ICPECVD (inductively coupled PECVD) is based on a new plasma source for high plasma density and specific fragmentation and precursor formation. The process operates at low pressures ( 5-20 Pa) with  $\text{SiH}_4$ ,  $\text{NH}_3$ , Ar and He as feed gases. In the table the main properties of ICPECVD layers deposited at 150 °C are compared with LPCVD, and PECVD.

The rather low etch rates of the ICPECVD layers in KOH as well as in buffered HF offer advantages in the MEMS technology. A low hydrogen concentration in the layer is responsible for these low etch rates.

process	Deposition temperature (°C)	Growth rate (nm/min)	Etch rate (BHF) (nm/min)	Etch rate (KOH) (nm/min)	Refractive index (632.8 nm)	Absorption index (320 nm)
LPCVD <sup>1</sup>	780	3.5	1.5 – 2	< 0.05 <sup>3</sup>	2.005	< 0.001
PECVD <sup>1</sup>	380	120 – 130	20 – 30	0.5 <sup>2</sup>	1.91 – 1.94	–
ICPECVD	150	50 – 60	3.5	< 0.1	2.0 – 2.08	0.019

<sup>1</sup> T.J.Cotler, J. Chapple-Sokol; J.el.chem.soc. 140 2071 (1993)

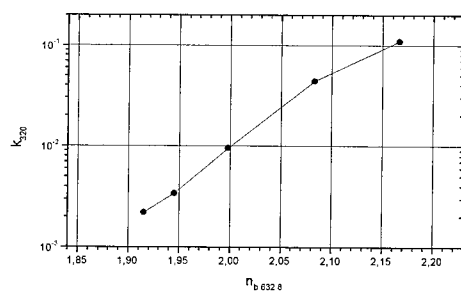
<sup>2</sup> Best value for ECR-PECVD

<sup>3</sup> Measured in direct comparison with IPECVD layers

An ideal value of 2,03 – 2,05 for the refractive index alone is not a figure of merit for silicon nitride layers. Otherwise, it is time consuming to measure the etch rates or even hydrogen concentrations.

A more complete and convenient method is to measure the absorption index in the

short wavelength range, e.g. at 320 nm. We found that layers with a refractive index near 2,03 and an absorption index below 0,01 have also excellent etch properties. From differently prepared layers as shown in the figure we can therefore select those with the best properties.



$n$  and  $k$  for layers prepared at different  $\text{SiH}_4 / \text{NH}_3$  flow ratio.

## Reliability requirements for microtechnologies used in automotive applications

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In this paper, the environmental and the lifetime requirements for electronics being used in automotive applications will be described, e.g. temperature, humidity and vibration. Such conditions and their specific damaging effects on components, assemblies and modules will be demonstrated. Furthermore trends and technologies for electronic systems for high reliability automotive applications will be described.

In automotive industry, electronics is a rapidly growing segment. This is demonstrated by the fact that the electrical equipment represents an increasing percentage of the fabrication costs. The actual market for vehicle electronics is about 11.3 bio US\$ and grows by approximately 16 % per year [1]. Table 1 shows the general development trends in automotive electronics. Legislation relating to the emissions from cars and other motor vehicles is driving the development of better combustion monitoring and control systems. Furthermore, improved safety levels are being achieved by the use of new types of electronic device for sensing and detecting the status of various aggregates, components and media (oil, gases, fuel) in a vehicle. Implementing electronically

controlled actuators leads to a much higher performance than current mechanical or hydraulic systems and enables on-board diagnosis, which will also become a legal demand. Benefits in terms of weight and complexity (i.e. cost) as well as reliability can be achieved if the electronics required for this is located close to the process being monitored. In many cases, the temperature can rise above 125°C, so that the use of high temperature electronic components is necessary.

Reliable operation of the elec-

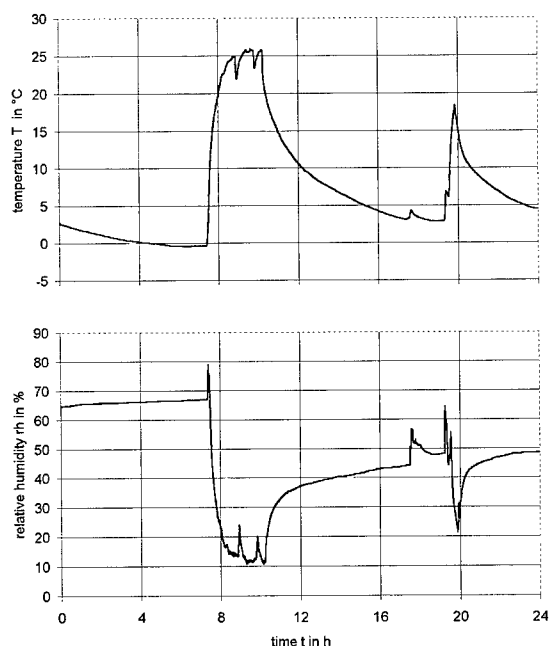


Figure 1:  
Environment in automotive applications: temperature and relative humidity in the passenger compartment on a mild winter day

Status	In production	Development status	Research status
Driver assistance	Cruise control	Adaptive cruise control	Traffic sign recognition
Vehicle dynamics	ESP ABS	Integrated chassis control	Drive by Wire
Traction/emissions	Engine controller	Power train control	Combustion control
Passenger protection	Crash detection airbag firing	Adaptive airbag control	Preocrash detection
Telematics	Navigation systems	Emergency assistance	Remote diagnosis

Table 1:  
Trends in the use of automotive electronics

Unit	Environmental Conditions		
	ECU	ECU	sensors
Environment Classification	»under the hood«	»on the engine«	»on the engine«
Temperature Range	-40 to 125 °C	-40 to 150 °C	-40 to 175 °C
Vibration	up to 3 g	up to 10 g	up to 40 g
Shock	up to 20 g	up to 30 g	up to 50 g

Table 2:  
Specification for use environments of automotive electronic units

tronics is of highest importance for these new applications. Measurements of the climatic conditions within the passenger compartment (see Fig. 1) demonstrate, that even in a protected zone of a car critical environmental conditions which potentially lead to failures can be observed. Environment specifications for electronic equipment are typically derived from such measurements taking into account the harshest conditions which can occur in service.

Table 2 lists such specifications depending on the location where the electronic unit is mounted in the car. Clearly the temperature ranges of on-the engine hardware which go up to 150 °C for ECUs and up to 175 °C for sensors exceed the specification limits of most electronic standard components. Direct mounting of electronics to the aggregates involves operation under harsh environmental conditions, like vibrations or high ambient temperatures, exceeding even the mil-specified limit of 125°C, and temperatures of even 200°C can be reached [2]. At the same time, longer lifetimes will be required for future automotive electronics.

This means hard requirements for electronic and micromechanical components. Some examples for the potential and the technological progress of different electronic components will be given.

[1] Financial Times Management Report »Automotive Electronics in Europe«, 1995

[2] W. Wondrak, Proceedings of the CARTS'99, pp. 123-126



### 3D-Microscope-ESPI: Potential for displacement analysis of micromechanic components

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Laser speckle interferometry as full field and non contact measuring technique offers highly interesting opportunities for deformation analysis on components. While its application in material testing and material research has already achieved an interesting level of acceptance in research and industry the application on very small components has been restricted. Present problems involve speckle size, optical access, decorrelation at object translation, etc. On the other hand, the potential of speckle interferometry to solve micromechanic measuring problems seems very high. Therefore, a new microscopic 3D-ESPI system has been developed. The system offers full field measurement capabilities on a measuring area of approximately 1 mm<sup>2</sup>. The local resolution of the system is better than 40 µm. Therefore, applications are possible on fairly small mechanical components. The system enables three-dimensional measurement of all components of the deformation vector on all surface points of the object. The 3D-Microscope-ESPI sensor itself is extremely miniaturized to enable easy access to the object's surface. Special focus was put on the simple operation without optical alignment. The paper describes the basic design and applications of the system on small scale areas on components.

## Numerical analysis of meniscus geometry and wetting force for wettability evaluation in reflow-mode wetting balance test

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The reflow-mode wetting balance test is a new approach to evaluate wettability of microelectronic materials for SMT technology such as solder paste, chip devices and PCB [1, 2]. Unlike conventional wetting balance test, solder metal is supplied as paste onto a micro pod, and geometric boundary constraint of liquid solder is complex and different in various sample shapes and dimensions. Therefore the meaning of force curve and its wettability parameters acquired by the new method are not fully well understood in the physical viewpoint.

To study this problem, extensive numerical analysis is conducted using Surface Evolver to realize meniscus geometry and wetting force in equilibrium wetting state of Pb-Sn eutectic solder liquid on copper rod models. Both calculated wetting height and force acted on specimen are increased with decreasing contact angle as for boundary condition. Those relationships are found not to be sensitive to dipping depth of specimen even under constant solder volume. Although dipping depth slightly influences overall geometry of solder meniscus, it drastically increases the maximum value of contact angle that could be formed at early stage of force curve just after solder melting, consequently non-wetting force value increases too. The above results show good agreement with experimental data. Proper interpretation of force curve and requirement of wettability parameter will be discussed at the conference.

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## Low cost bumping on waferlevel for 300 mm wafers

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This paper presents the requirements for electroless Ni / Au bumping for 300 mm Wafers. The equipment and process requirements together with first results of bumping on 12 inch wafers are presented.

A summary and outlook on next generation wafers using copper metallization is presented.

## Condensation and nucleation of noble metals on low-k polymers

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The structure and properties of metal-polymer interfaces depend strongly on the deposition conditions. Particularly the condensation and nucleation behavior in the early deposition process play a crucial role. By using a novel very sensitive radiotracer technique and photoelectron spectroscopy we have determined condensation coefficients  $C$  of noble metals on several polymers.  $C$  is defined as the ratio of the number of adsorbed atoms to the total number of metal atoms arriving at the surface.  $C$  shows an extreme variation ranging from close to unity for polyimide to values as low as 0.002 for Ag on Teflon AF<sup>TM</sup> at room temperature. The extreme variation in the condensation behavior upon different polymers appears to be related to the polymer surface energy and suggests a connection between macroscopic wetting and atomic condensation. At elevated temperatures the condensation coefficient decreases strongly on all polymers. The angular distribution of reemitted atoms has a  $\cos\theta$  form and does not depend on the angle of incidence. This, among other arguments, suggests them not to be scattered directly but to be adsorbed at the surface prior to reemission. In some cases such as Teflon AF<sup>TM</sup> nucleation turned out to take place at preferred sites, as reflected in a condensation coefficient being independent of the evaporation rate. The nature of these sites is not known yet; one can think of terminal groups on the polymer chains, impurities, or attractive local arrangements of the chains. The site density, and thus the condensation coefficient, were found to increase substantially upon ion beam treatment. At sufficiently high metal coverages the condensation coefficient always approaches unity, as expected for metal-on-metal condensation. Random nucleation with a critical nucleus consisting of only one atom was observed for noble metals on polyimide. Adsorption energies and activation energies of surface diffusion were determined from the temperature and deposition-rate dependence of the condensation coefficient and the maximum cluster density.

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